#### PROCESS TO PREPARE A LUBRICATING BASE OIL

The invention is directed to a process to prepare a base oil having an paraffin content of between 75 and 95 wt%.

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WO-A-0246333 describes a process to prepare two viscosity grades of base oil by solvent dewaxing a fraction having a T95% point above 621 °C and catalytically dewaxing a fraction having a T95% point of below 621 °C. The two fractions are Fischer-Tropsch derived fractions. Optionally the heavier or the lower boiling fraction may also be a slack wax, a distillate from crude oil, deasphalted residual stocks from crude oil.

NL-C-1015035 describes a process to prepare a base oil from a Fischer-Tropsch derived feed by performing a hydroisomerisation step. The effluent of the hydroisomerisation step is distilled and a residue boiling above 380 °C is obtained. This residue is subjected to a catalytic dewaxing treatment using a catalyst containing platinum and ferrierite.

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US-A-6294077 describes a catalytic dewaxing treatment wherein a catalyst is used consisting of ZSM-5 and platinum.

US-A-6025305 discloses a process wherein a Fischer-Tropsch wax feed is first hydroisomerised. The effluent of the hydroisomerisation is then separated into fuels and lubricants. No pour point reducing treatment is disclosed in this publication.

US-A-2002/0146358 describes a process for hydroisomerisation of a Fischer-Tropsch derived wax feed. The effluent of the hydroisomerisation step is distilled and a bottoms fraction comprising compounds having 20 or

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more carbon atoms is obtained. This bottoms fraction may be subjected to a catalytic dewaxing treatment.

WO-A-0157166 describes the use of a highly paraffinic base oil as obtained from a Fischer-Tropsch wax in a motor engine lubricant formulation. The examples illustrate that such formulations will also consist of an ester, which according to the description of the patent are added to confer additional desired characteristics, such as additive solvency.

The use of ester co-base fluids in lubricant formulations as illustrated in WO-A-0157166 is not desired because such ester co-base fluids are not widely available and thus expensive. Additive solvency may be improved by using a paraffinic base stock, which contains less paraffins. Such base oils may be prepared by hydroisomerisation of petroleum derived waxes followed by a solvent or catalytic dewaxing step. A disadvantage of such a process is that the starting petroleum derived waxes, such as for example slack wax, are not easily obtainable. Furthermore such waxes may not always have the desired high paraffin content needed to make the desired base oils as per this invention.

The object of the present invention is to provide a process wherein a base oil with a paraffin content of between 75 and 95 wt% is obtained which does not have the disadvantages of the prior art processes.

This object is achieved by the following process.

Process to prepare a base oil having an paraffin content of between 75 and 95 wt% by subjecting a mixture of a Fischer-Tropsch derived feed and a petroleum derived feed to a catalytic pour point reducing treatment.

Applicants found that by mixing a relatively small amount of a petroleum derived feed with a Fischer-Tropsch derived feed before performing a catalytic pour point

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reducing treatment a base oil may be obtained having the desired properties.

The petroleum-derived fraction may in principle be any fraction boiling in the base oil range and containing non-paraffinic compounds. For this reason the wax content of such a feed will be typically low because wax typically consists for a greater part of normal paraffins. The wax content is suitably lower than 50 wt% and preferably lower than 30 wt%, as measured at -27 °C using a 50/50 (vol/vol) methyl-ethyl ketone/toluene solvent mixture. In an even more preferred embodiment the wax content is very low and the feed is better described by its low pour point of below -10 °C and more preferably below -15 °C.

Preferably a petroleum-derived fraction is used which has been subjected to a hydroprocessing step in order to reduce aromatic, sulphur and nitrogen content of such fractions and improve some of the desired properties such viscosity index. The hydroprocessing step may be a hydrotreating optionally followed by a hydrocracking step. Such processes are for example performed when preparing base oils from a petroleum derived vacuum distillate or de-asphalted oils.

The petroleum derived feed preferably has an aromatic compound content of between 0 and 20 wt% and a naphthenic compound content of preferably between 15 and 90 wt%. These contents can be measured by well-known techniques such as the technique described at Example 3 of this specification.

A very interesting petroleum derived feed is the bottoms fraction of a fuels hydrocracker. With a fuels hydrocracker in the context of the present invention is meant a hydrocracker process which main products are naphtha, kerosene and gas oil. The conversion, expressed in the weight percentage of the fraction in the feed to

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the hydrotreater-hydrocracker which boils above 370 °C which are converted to products boiling below 370 °C, in the hydrotreater-hydrocracker process is typically above 50 wt%. Examples of possible fuels hydrocracker processes, which may yield a bottoms fraction which can be used in the present process, are described in the above referred to EP-A-699225, EP-A-649896, WO-A-9718278, EP-A-705321, EP-A-994173 and US-A-4851109.

Another interesting petroleum derived feed is the fraction obtained in a dedicated base oil hydrotreater-hydrocracker. In such a hydrotreater-hydrocracker the main products will boil in the base oil range. Typically such processes operate at a feed conversion of below 50 wt% and more typically between 20 and 40 wt%. The petroleum derived feed is thus the high boiling fraction as obtained in such a process prior to dewaxing.

Preferably the fuels hydrocracker is operated in two steps, consisting of a preliminary hydrotreating step followed by a hydrocracking step. In the hydrotreating step nitrogen and sulphur are removed and aromatics are saturated to naphthenes

An even more preferred feedstock is a dewaxed oil. This oil preferably has a pour point of below -10 °C, more preferably below -15 °C. Dewaxing can be solvent or catalytic dewaxing. The saturates content is preferably greater than 90 wt%, more preferably greater than 95 wt% and even more preferably greater than 98 wt% and most preferably higher than 99 wt%. The sulphur content is preferably less than 0.03 wt%, more preferably less than 0.01 wt% and even more preferably less than 0.01 wt%. An advantage of using a dewaxed oil having such low contents of sulphur, nitrogen and high contents of saturates is that no additional hydrofinishing is required after performing the pour point reducing step of the process of the present

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invention. Advantageously the catalytic dewaxing will then not have to be performed at the higher pressure required for the subsequent hydrofinishing. In contrast it can be performed at a more preferred lower hydrogen pressure range of between 40 and 70 bars. Omitting such a hydrofinishing step is especially possible if the mineral derived dewaxed oils themselves are prepared by means of a process which does include a hydrofinishing step, preferably performed at a hydrogen pressure of above 100 bars. Examples of such hydrofinishing processes are for example those described below.

The oils are preferably made starting from a vacuum distillate or a de-asphalted vacuum residue of a mineral crude oil feedstock or from a waxy feed such as a slack wax, wherein the process includes a hydroprocessing step in which the sulphur and polar compounds are reduced to the preferred ranges. The viscosity index is preferably between 80-150, while good results have been achieved with oils having a viscosity index of between 80 and 120.

Preferably the T10wt% recovery point of this oil is between 200 and 450 °C, more preferably between 300 and 420 °C and the T90wt% recovery point is between 300 and 550 °C, more preferably between 400 and 550 °C. By using such a wide boiling oil it has been found possible to reduce the iso-paraffin content of the resultant base oils for both the lower viscosity grades, ranging from 2 cSt at 100 °C kinematic viscosity, to and including the higher viscosity grades having a kinematic viscosity at 100 °C of 15 cSt.

The dewaxed oil can be obtained as such by well known processes as for example described in Chapter 6 of Lubricant Base Oil and Wax Processing, Avilino Sequeira, Jr, Marcel Dekker, Inc. New York, 1994, page 119-150. The preferred wide boiling oil can be prepared by mixing various viscosity grades of, preferably API Group II or

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Group III, base oils. Examples of processes which yield an oil which can be used in this process are described in EP-A-0909304, EP-A-1137741, EP-A-1392799, EP-A-1311651. Examples of suitable dewaxed oils are for example Shell's XHVI-4, XHVI-5.2 and XHVI-8 base oil products or ExxonMobil's Visom base oil grades and mixtures thereof. A possible commercial process which yields base oil for use in this invention is ExxonMobil's MSDW<sup>TM</sup>/MAXSAT<sup>TM</sup> type of process which is said to yield a base oil containing less than 1 wt% aromatics, < 1ppm sulphur, a Viscosity Index of greater than 120 and a pour point of less than -15 °C.

A more preferred dewaxed oil, which meets the above description, are those obtained when the bottoms fraction of a fuels hydrocracker, as described above, is catalytically dewaxed followed by a hydrofinishing step. Examples of publications describing this route are WO-A-9802502, WO-A-0027950, WO-A-9500604, EP-A-0883664 and EP-A-0863963.

An additional advantage of adding a dewaxed oil as described above to the pour point reducing treatment of the process of the invention is that any undesired compounds in said oil, such as for example wax, polars, sulphur or nitrogen, can be further reduced in said treatment. A further advantage is that the boiling range properties, pour point and/or the volatility of the final base oil can be controlled in a simple manner by control of the dewaxing conditions and the optional further distillation of the product obtained in said dewaxing treatment. This is advantageous because it makes possible to use a wide variation of dewaxed oils in the process according to the invention. If for example such oils would have been blended after the dewaxing of a 100% Fischer-Tropsch derived feed and after a final distillation much stringent property specifications, like

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for example Noack volatility and viscosity, for the mineral derived blending component would have been required. Thus the process according the invention makes it possible to use a wide variation of dewaxed oils having the above properties and obtain a base oil having the desired paraffin content and other desired base oil properties like especially Noack volatility and pour point.

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The Fischer-Tropsch derived feed preferably is a hydroisomerized Fischer-Tropsch wax. Such a feed may be obtained by well-known processes, for example the so-called commercial Sasol process, the Shell Middle Distillate Process or by the non-commercial Exxon process. These and other processes are for example described in more detail in EP-A-776959, EP-A-668342, US-A-4943672, US-A-5059299, WO-A-9934917 and WO-A-9920720. The process will generally comprise a Fischer-Tropsch synthesis and a hydroisomerisation step as described in these publications. Such a process to prepare the hydroisomerised Fischer-Tropsch feed for use as feed in the present process will comprise the following steps: (a) hydrocracking/hydroisomerisating a Fischer-Tropsch product,

(b) separating by means of distillation the product of step (a) into one or more gas oil fractions and a higher boiling Fischer-Tropsch derived feed according to this invention.

Preferably the Fischer-Tropsch product used as feed in step (a) is a product wherein the weight ratio of compounds having at least 60 or more carbon atoms and compounds having at least 30 carbon atoms in the Fischer-Tropsch product is at least 0.2 and wherein at least 30 wt% of compounds in the Fischer-Tropsch product have at least 30 carbon atoms.

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Applicants found that by performing the hydrocracking/hydroisomerisation step with the relatively heavy feedstock a higher yield of gas oils as calculated on the feed to step (a) can be obtained. A further advantage is that both fuels, for example gas oil, and the Fischer-Tropsch derived feed are prepared in one hydrocracking/hydroisomerisation process step. In a preferred embodiment of the present invention a fraction boiling above the Fischer-Tropsch derived feed is isolated in step (b) and recycled to step (a).

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A further advantage is that by performing step (a) on the relatively heavy feed a Fischer-Tropsch derived feed is prepared which already has a certain content of cycloparaffins.

The relatively heavy Fischer-Tropsch product used in step (a) has more preferably at least 50 wt%, and even more preferably at least 55 wt% of compounds having at least 30 carbon atoms. Furthermore the weight ratio of compounds having at least 60 or more carbon atoms and compounds having at least 30 carbon atoms of the Fischer-Tropsch product is more preferably at least 0.4 and even more preferably at least 0.50. Preferably the Fischer-Tropsch product comprises a  $C_{20}^+$  fraction having an ASF-alpha value (Anderson-Schulz-Flory chain growth factor) of at least 0.925, preferably at least 0.935, more preferably at least 0.945, even more preferably at least 0.955.

The initial boiling point of the Fischer-Tropsch product may range up to 400 °C, but is preferably below 200 °C. Preferably any compounds having 4 or less carbon atoms and any compounds having a boiling point in that range are separated from a Fischer-Tropsch synthesis product before the Fischer-Tropsch synthesis product is used in step (a). The Fischer-Tropsch product as described in detail above is a Fischer-Tropsch product,

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which has not been subjected to a hydroconversion step as defined according to the present invention. The content of non-branched compounds in the Fischer-Tropsch product will therefore be above 80 wt%. In addition to the Fischer-Tropsch product also other fractions may be additionally processed in step (a). Possible other fractions may suitably be the optional higher boiling fraction obtained in step (b) or part of said fraction and/or off-spec base oil fractions as obtained in the pour point reducing treatment of the process of the present invention.

Such a Fischer-Tropsch product can be obtained by any process, which yields a relatively heavy Fischer-Tropsch product as described above. Not all Fischer-Tropsch processes yield such a heavy product. An example of a suitable Fischer-Tropsch process is described in WO-A-9934917 and in AU-A-698392. These processes may yields a Fischer-Tropsch product as described above.

The Fischer-Tropsch product will contain no or very little sulphur and nitrogen containing compounds. This is typical for a product derived from a Fischer-Tropsch reaction, which uses synthesis gas containing almost no impurities. Sulphur and nitrogen levels will generally be below the detection limits, which are currently 2 ppm for sulphur and 1 ppm for nitrogen respectively.

The Fischer-Tropsch product may optionally be subjected to a mild hydrotreatment step in order to remove any oxygenates and saturate any olefinic compounds present in the reaction product of the Fischer-Tropsch reaction. Such a hydrotreatment is described in EP-B-668342. The mildness of the hydrotreating step is preferably expressed in that the degree of conversion in this step is less than 20 wt% and more preferably less than 10 wt%. The conversion is here defined as the weight percentage of the feed boiling above 370 °C, which reacts

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to a fraction boiling below 370 °C. After such a mild hydrotreatment lower boiling compounds, having four or less carbon atoms and other compounds boiling in that range, will preferably be removed from the effluent before it is used in step (a).

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The hydrocracking/hydroisomerisation reaction of step (a) is preferably performed in the presence of hydrogen and a catalyst, which catalyst can be chosen from those known to one skilled in the art as being suitable for this reaction of which some will be described in more detail below. The catalyst may in principle be any catalyst known in the art to be suitable for isomerising paraffinic molecules. In general, suitable hydroconversion catalysts are those comprising a hydrogenation component supported on a refractory oxide carrier, such as amorphous silica-alumina, alumina, fluorided alumina, molecular sieves (zeolites) or mixtures of two or more of these. One type of preferred catalysts to be applied in the hydroconversion step in accordance with the present invention are hydroconversion catalysts comprising platinum and/or palladium as the hydrogenation component. A very much preferred hydroconversion catalyst comprises platinum and palladium supported on an amorphous silica-alumina (ASA) carrier. The platinum and/or palladium is suitably present in an amount of from 0.1 to 5.0% by weight, more suitably from 0.2 to 2.0% by weight, calculated as element and based on total weight of carrier. If both present, the weight ratio of platinum to palladium (calculated as element) may vary within wide limits, but suitably is in the range of from 0.05 to 10, more suitably 0.1 to 5. Examples of suitable noble metal on ASA catalysts are, for instance, disclosed in WO-A-9410264 and EP-A-0582347. Other suitable noble metal-based catalysts, such as platinum on a fluorided alumina carrier, are disclosed in

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e.g. US-A-5059299 and WO-A-9220759. Preferably such catalysts do not comprise a molecular sieve, more preferably such catalysts do not comprise zeolite beta.

A second type of suitable hydroconversion catalysts are those comprising at least one Group VIB metal, preferably tungsten and/or molybdenum, and at least one non-noble Group VIII metal, preferably nickel and/or cobalt, as the hydrogenation component. Usually both metals are present as oxides, sulphides or a combination thereof. The Group VIB metal is suitably present in an amount of from 1 to 35% by weight, more suitably from 5 to 30% by weight, calculated as element and based on total weight of catalyst. The non-noble Group VIII metal is suitably present in an amount of from 1 to 25 %wt, preferably 2 to 15 %wt, calculated as element. and based on total weight of carrier. A hydroconversion catalyst of this type which has been found particularly suitable is a catalyst comprising nickel and tungsten supported on fluorided alumina.

A preferred catalyst which can be used in a non-sulphided form comprises a non-noble Group VIII metal, e.g., iron, nickel, in conjunction with a Group IB metal, e.g., copper, supported on an acidic support. The catalyst has a surface area in the range of 200-500 m<sup>2</sup>/gm, preferably 0.35 to 0.80 ml/gm, as determined by water adsorption, and a bulk density of about 0.5-1.0 g/ml. The catalyst support is preferably an amorphous silica-alumina where the alumina is present in amounts of less than about 30 wt%, preferably 5-30 wt%, more preferably 10-20 wt%. Also, the support may contain small amounts, e.g., 20-30 wt%, of a binder, e.g., alumina, silica, Group IVA metal oxides, and various types of clays, magnesia, etc., preferably alumina.

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The preparation of amorphous silica-alumina microspheres has been described in Ryland, Lloyd B., Tamele, M.W., and Wilson, J.N., Cracking Catalysts, Catalysis: volume VII, Ed. Paul H. Emmett, Reinhold Publishing Corporation, New York, 1960, pp. 5-9.

The catalyst is prepared by co-impregnating the metals from solutions onto the support, drying at 100-150 °C, and calcining in air at 200-550 °C. The Group VIII metal is present in amounts of about 15 wt% or less, preferably 1-12 wt%, while the Group IB metal is usually present in lesser amounts, e.g., 1:2 to about 1:20 weight ratio respecting the Group VIII metal.

A typical catalyst is shown below:

	Ni, wt%	2.5-3.5
15	Cu, wt%	0.25-0.35
	Al <sub>2</sub> O <sub>3</sub> -SiO2 wt%	65- 75
	Al <sub>2</sub> O <sub>3</sub> (binder) wt%	25-30
	Surface Area	290-325 m <sup>2</sup> /gm
	Pore Volume (Hg)	0.35-0.45 ml/gm

Bulk Density

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Another class of suitable hydroconversion catalysts are those based on zeolitic materials, suitably comprising at least one Group VIII metal component, preferably Pt and/or Pd, as the hydrogenation component.

Suitable zeolitic materials, then, include Zeolite beta, Zeolite Y, Ultra Stable Y, ZSM-5, ZSM-12, ZSM-22, ZSM-23, ZSM-35, SSZ-32, ferrierite, zeolite beta, mordenite and

silica-aluminophosphates, such as SAPO-11 and SAPO-31.

0.58 - 0.68 g/ml

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Examples of suitable hydroisomerisation catalysts are, for instance, described in WO-A-9201657.

In step (a) the feed is contacted with hydrogen in the presence of the catalyst at elevated temperature and pressure. The temperatures typically will be in the range of from 175 to 380 °C, preferably higher than 250 °C and more preferably from 300 to 370 °C. The pressure will typically be in the range of from 10 to 250 bar and preferably between 20 and 80 bar. Hydrogen may be supplied at a gas hourly space velocity of from 100 to 10000 Nl/l/hr, preferably from 500 to 5000 Nl/l/hr. The hydrocarbon feed may be provided at a weight hourly space velocity of from 0.1 to 5 kg/l/hr, preferably higher than 0.5 kg/l/hr and more preferably lower than 2 kg/l/hr. The ratio of hydrogen to hydrocarbon feed may range from 100 to 5000 Nl/kg and is preferably from 250 to 2500 Nl/kg.

The conversion in step (a) as defined as the weight percentage of the feed boiling above 370 °C which reacts per pass to a fraction boiling below 370 °C, is at least 20 wt%, preferably at least 25 wt%, but preferably not more than 80 wt%, more preferably not more than 70 wt%. The feed as used above in the definition is the total hydrocarbon feed fed to step (a), thus also any optional recycle of the higher boiling fraction as obtained in step (b).

In step (b) the product of step (a) is separated into one or more gas oil fractions and a Fischer-Tropsch derived feed having preferably a T10 wt% boiling point of between 200 and 450 °C. If a higher boiling fraction is separated from the Fischer Tropsch feed the T90 wt% of said feed is preferably between 300 °C, and preferably between 430 and 550 °C. The separation is preferably performed by means of a first distillation at about atmospheric conditions, preferably at a pressure of

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between 1.2-2 bara, wherein the gas oil product and lower boiling fractions, such as naphtha and kerosine fractions, are separated from the higher boiling fraction of the product of step (a). The higher boiling fraction, of which suitably at least 95 wt% boils above 370 °C, may be further separated in a vacuum distillation step wherein a higher boiling fraction is separated. The vacuum distillation is suitably performed at a pressure of between 0.001 and 0.05 bara.

The vacuum distillation of step (b) is preferably operated such that the desired Fischer Tropsch derived feed is obtained boiling in the specified range and having a kinematic viscosity, which relates to the base oil end product(s) specification. The kinematic viscosity at 100 °C of the Fischer Tropsch derived feed is preferably between 3 and 10 cSt.

The mixture of petroleum derived and Fischer-Tropsch derived feeds will suitably have a viscosity corresponding to the desired viscosity of the base oil product. Preferably the kinematic viscosity at 100 °C of the mixture is between 3 and 10 cSt. Suitable distillate fractions have a T10wt% boiling point of between 200 and 450 °C, preferably between 300 and 420 °C and a T90wt% boiling point of between 300 and 550 °C, preferably between 400 and 550 °C. The fraction of petroleum derived feed in the mixture is preferably higher than 5 wt%, more preferably higher than 10 wt% and preferably lower than 50 wt% and more preferably below 30 wt% and even more preferably below 25 wt%. The actual content of petroleum-derived feed in the mixture will of course depend on the paraffin content of said feed. The mixture will preferably contain less than 50 ppm sulphur and/or less that 10 ppm nitrogen.

With the catalytic pour point reducing treatment is understood every process wherein the pour point of the

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base oil is reduced by more than 10 °C, preferably more than 20 °C, more preferably more than 25 °C.

The catalytic dewaxing or pour point reducing process can be performed by any process wherein in the presence of a catalyst and hydrogen the pour point of the mixture is reduced as specified above. Suitable dewaxing catalysts are heterogeneous catalysts comprising a molecular sieve and optionally in combination with a metal having a hydrogenation function, such as the Group VIII metals. Molecular sieves, and more suitably intermediate pore size zeolites, have shown a good catalytic ability to reduce the pour point of the distillate base oil precursor fraction under catalytic: dewaxing conditions. Preferably the intermediate pore size zeolites have a pore diameter of between 0.35 and 0.8 nm. Suitable intermediate pore size zeolites are mordenite, ZSM-5, ZSM-12, ZSM-22, ZSM-23, SSZ-32, ZSM-35 and ZSM-48. Another preferred group of molecular sieves are the silica-aluminaphosphate (SAPO) materials of which SAPO-11 is most preferred as for example described in US-A-4859311. ZSM-5 may optionally be used in its HZSM-5 form in the absence of any Group VIII metal. The other molecular sieves are preferably used in combination with an added Group VIII metal. Suitable Group VIII metals are nickel, cobalt, platinum and palladium. Examples of possible combinations are Ni/ZSM-5, Pt/ZSM-23, Pd/ZSM-23, Pt/ZSM-48 and Pt/SAPO-11. Further details and examples of suitable molecular sieves and dewaxing conditions are for example described in WO-A-9718278, US-A-5053373, US-A-5252527 and US-A-4574043.

The dewaxing catalyst suitably also comprises a binder. The binder can be a synthetic or naturally occurring (inorganic) substance, for example clay, silica and/or metal oxides. Natural occurring clays are for example of the montmorillonite and kaolin families. The

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binder is preferably a porous binder material, for example a refractory oxide of which examples are: alumina, silica-alumina, silica-magnesia, silica-zirconia, silica-thoria, silica-beryllia, silica-titania as well as ternary compositions for example silica-alumina-thoria, silica-alumina-zirconia, silica-alumina-magnesia and silica-magnesia-zirconia. More preferably a low acidity refractory oxide binder material which is essentially free of alumina is used. Examples of these binder materials are silica, zirconia, titanium dioxide, germanium dioxide, boria and mixtures of two or more of these of which examples are listed above. The most preferred binder is silica.

A preferred class of dewaxing catalysts comprise intermediate zeolite crystallites as described above and a low acidity refractory oxide binder material which is essentially free of alumina as described above, wherein the surface of the aluminosilicate zeolite crystallites has been modified by subjecting the aluminosilicate zeolite crystallites to a surface dealumination treatment. A preferred dealumination treatment is by contacting an extrudate of the binder and the zeolite with an aqueous solution of a fluorosilicate salt as described in for example US-A-5157191 or WO-A-2000029511. Examples of suitable dewaxing catalysts as described above are silica bound and dealuminated Pt/ZSM-5, silica bound and dealuminated Pt/ZSM-23, silica bound and dealuminated Pt/ZSM-12, silica bound and dealuminated Pt/ZSM-22 as for example described in WO-A-200029511 and EP-B-832171.

Catalytic dewaxing conditions are known in the art and typically involve operating temperatures in the range of from 200 to 500 °C, suitably from 250 to 400 °C, hydrogen pressures in the range of from 10 to 200 bar, preferably from 40 to 70 bar, weight hourly space

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velocities (WHSV) in the range of from 0.1 to 10 kg of oil per litre of catalyst per hour (kg/l/hr), suitably from 0.2 to 5 kg/l/hr, more suitably from 0.5 to 3 kg/l/hr and hydrogen to oil ratios in the range of from 100 to 2,000 litres of hydrogen per litre of oil. By varying the temperature between 315 and 375 °C at a pressure of between 40-70 bars, in the catalytic dewaxing step it is possible to prepare base oils having different pour point specifications varying from suitably lower than -60 to -10 °C.

Optionally a noble metal guard bed may be positioned just upstream the dewaxing step, for example as a separate catalyst bed in the dewaxing reactor. Such a guard bed is advantageous to remove any remaining sulphur and especially nitrogen compounds present in the feed to the dewaxing process of the present invention. Such a guard bed is suitably used when a bottoms fraction of a fuels hydrocracker process is used as the petroleum derived feed. An example of such a process is described in WO-A-9802503, which reference is hereby incorporated by reference.

After performing the pour point reducing treatment lower boiling compounds formed during said treatment are suitably removed, preferably by means of distillation, optionally in combination with an initial flashing step.

The effluent of the pour point reducing treatment may suitably be subjected to a hydrogenation treatment. Hydrogenation may be performed on the entire effluent or on specific base oil grades after the above described fractionation. This may be required in order to reduce the content of aromatic compounds in the reduced pour point product to preferably values of below 1 wt%. Such a hydrogenation is also referred to as a hydrofinishing step. This step is suitably carried out at a temperature between 180 and 380 °C, a total pressure of

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between 10 to 250 bar and preferably above 100 bar and more preferably between 120 and 250 bar. The WHSV (Weight hourly space velocity) ranges from 0.3 to 2 kg of oil per litre of catalyst per hour (kg/l.h). Preferably a hydrogenation is performed in the same reactor as the catalytic dewaxing reactor. In such a reactor the beds of dewaxing catalyst and hydrogenation catalyst will be placed in a stacked bed on top of each other.

The hydrogenation catalyst is suitably a supported catalyst comprising a dispersed Group VIII metal.

Possible Group VIII metals are cobalt, nickel, palladium and platinum. Cobalt and nickel containing catalysts may also comprise a Group VIB metal, suitably molybdenum and tungsten. Suitable carrier or support materials are low acidity amorphous refractory oxides. Examples of suitable amorphous refractory oxides include inorganic oxides, such as alumina, silica, titania, zirconia, boria, silica-alumina, fluorided alumina, fluorided silica-alumina and mixtures of two or more of these.

Examples of suitable hydrogenation catalysts are nickel-molybdenum containing catalyst such as KF-847 and KF-8010 (AKZO Nobel) M-8-24 and M-8-25 (BASF), and C-424, DN-190, HDS-3 and HDS-4 (Criterion); nickel-tungsten containing catalysts such as NI-4342 and NI-4352 (Engelhard) and C-454 (Criterion); cobalt-molybdenum containing catalysts such as KF-330 (AKZO-Nobel), HDS-22 (Criterion) and HPC-601 (Engelhard). Preferably platinum containing and more preferably platinum and palladium containing catalysts are used. Preferred supports for these palladium and/or platinum containing catalysts are amorphous silica-alumina. Examples of suitable silicaalumina carriers are disclosed in WO-A-9410263. A preferred catalyst comprises an alloy of palladium and platinum preferably supported on an amorphous silicaalumina carrier of which the commercially available

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catalyst C-624 of Criterion Catalyst Company (Houston, TX) is an example.

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After performing the catalytic pour point reducing treatment or after the optional hydrofinishing step hydrogen is suitably separated from the dewaxed/hydrofinished effluent, contacted with a means to remove hydrogen sulphide and recycled to said catalytic pour point reducing treatment. Such means could be amine washing of the hydrogen recycle stream. If the content of hydrogen sulphide is expected to be low, for example below 100 ppm or even below 20 ppm, in the recycle stream contacting said stream with a suitable adsorbent will be preferred. Examples of suitable heterogeneous adsorbents are comprise at least one metal or oxide of the metal, the metal(s) being selected from Fe, Ni, Co, Ag, Sn Re, Mo, Cu, Pt, Pd and Zn. In a preferred embodiment, the metal is at least one of Fe, Ni, Co, Cu, and Zn. In a more preferred embodiment the adsorbent is zinc oxide. The adsorbent may be supported on an inorganic support material in order to, for example, increase surface area, pore volume, and pore diameter. Suitable support materials contain at least one inorganic refractory support materials including, but not necessarily limited to, alumina, silica, zirconia, carbon, silicon carbide, kieselguhr, amorphous and crystalline silica-aluminas, silica-magnesias, aluminophosphates, boria, titania, and zinc oxide. Preferred support materials include alumina, zirconia, and silica. The metal(s) or metal oxide(s) may be loaded onto these supports by conventional techniques known in the art. Non-limiting examples of suitable supported metal and metal oxide based regenerable sulfur adsorbents include, but are not necessarily limited to: Co/Al<sub>2</sub>O<sub>3</sub>; Co/SiO<sub>2</sub>; Co/TiO<sub>2</sub>; Co/ZrO<sub>2</sub>; Ni/Al<sub>2</sub>O<sub>3</sub>; Ni/SiO<sub>2</sub>; Ni/ZrO2; Cu/Al2O3; CU/SiO2; Cu/ZrO2; Fe/Al2O3; Fe/SiO2; Fe/ZrO2; Co/Cu/Al2O3; Co/Cu/SiO2; Ni/Cu/SiO2; Ni/Cu/ZrO2;

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Co/Pt/Al<sub>2</sub>O<sub>3</sub>; Co/Pd/SiO<sub>2</sub>; Co/Sn/Al<sub>2</sub>O<sub>3</sub>; Ni/Sn/SiO<sub>2</sub>; Zn/Al<sub>2</sub>O<sub>3</sub>, ZnO/SiO<sub>2</sub>, Co/ZnO; Mo/ZnO; Ni/ZnO; Co/Mo/ZnO; Ni/Mo/ZnO; Pt/ZnO; Pt/Pd/ZnO. The adsorbent may also be employed as a bulk metal oxide or as a bulk metal, including but not necessarily limited to, a finely divided skeleton metal, including Raney metals, ponderous metals, Rieke metals, and metal sponges. The temperature and pressure conditions during said contacting are preferably within the ranges specified for the catalytic pour point reduction.

From the effluent of the pour point reducing treatment and the optional hydrogenation treatment one or more base oil grades may be isolated by means of fractionation. Base oil products having kinematic viscosity at 100 °C of between 2 and 10 cSt, having a volatility of between 8 and 11% (according to CEC L40 T87) and a pour point of between -20 and -60 °C (according to ASTM D 97) may advantageously be obtained.

The content of paraffins is more preferably less than 90 wt% and more preferably higher than 80 wt%.

The above-described base oil can suitably find use as base oil for an Automatic Transmission Fluids (ATF), motor engine oils, electrical oils or transformer oils and refrigerator oils. lubricant formulations such as motor engine oils of the OW-x and 5W-x specification according to the SAE J-300 viscosity classification, wherein x is 20, 30, 40, 50 or 60 may be advantageously made using this base oil.

It has been found that lubricant formulations can be prepared with the base oils obtainable by the process of the current invention without the need to add high contents of additional ester or aromatic co-base oils. Preferably less than 15 wt% and more preferably less than 10 wt% of such ester or aromatic co-base oil is present in such formulations.

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The Fischer-Tropsch process is sometimes performed at a remote location far away from the end-users of the base oils. It has also been found that for certain applications the end-users do not necessarily require base oils having the high paraffin contents as prepared by the prior art processes which operate on 100 % Fischer-Tropsch derived feed. For these applications blending with mineral derived base oils containing less paraffins will have to take place such to reduce the paraffin content. It is however not always the case that suitable mineral blending components are found near the end users. As explained above such blending components need to have the right volatility and viscosity to obtain the desired blend. The present invention solves this problem, wherein at the remote location the desired lower paraffin base oils are prepared having the specified volatility and viscosity and in addition pour point and viscosity index from a petroleum derived feed which does not have to meet all the stringent quality properties. Thus a process is obtained wherein for example the petroleum derived feed is obtained from one location and the base oils obtained by the present process are marketed in many different locations. In a preferred embodiment the petroleum derived feed is shipped to the remote location from another location and part of the base oils as made by the present process are shipped to said other location making use of the same vessel. This is advantageous because efficient use of shipping capacity between the two locations is thus achieved. Such a method is especially suitable when the petroleum derived feed is a dewaxed oil having the preferred low sulphur contents as described above.

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#### Example 1 and 2

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A blend of 20 wt% of a bottoms fraction of a fuels hydrocracker and 80 wt% of a Fischer-Tropsch derived Waxy Raffinate as obtained from Shell MDS (Malaysia) Sdn Bhd (marketed as Shell MDS Waxy Raffinate) was prepared. The Shell MDS Waxy Raffinate is a partly isomerised Fischer-Tropsch derived fraction.

The above blend was contacted with a dewaxing catalyst consisting of 0.7 wt% platinum, 25 wt% ZSM-12 and a silica binder. The dewaxing conditions were 40 bar hydrogen, WHSV = 1 kg/l.h, and a hydrogen gas rate of 700 Nl/kg feed. The experiment was carried out at two different reaction temperatures, namely 304 °C(Example 1) and 297 °C (Example 2).

From the effluent of the dewaxing step a base oil boiling between 400 and 460 °C was isolated from lower and higher boiling products. This fraction was analysed. The yield and properties of this fraction having a kinematic viscosity of about 4 cSt are reported in Table 2. Table 5 lists the composition of these base oils.

Table 1

Feed	bottoms fraction of a fuels hydrocracker	Shell MDS Waxy Raffinate
Density (D70/4)	0.8036	0.7845
Pour point°C	+39	+42°C
Nitrogen (ppmw)	< 1	n.a.(*)
Sulphur (ppmw)	< 2	n.a.(*)

Kinematic		
viscosity	4.439	5.062
at 100 °C		
cSt		
Initial	, <u> </u>	
boiling	269	361
point	269	361
(IBP) °C		
IBP-390	26.9	4.4
°C (wt%)	20.9	77 - 73
390-520	63.0	84.8
°C (wt%)	05.0	04.0
520 °C −	10.1	10.8
FBP (wt%)	10.1	
Final		
boiling	602	587
point	1 002	
(FBP) °C		
Wax		16.4
content	17.8	
(wt%)		

\* N.a. = not analysed. In view of Fischer-Tropsch source it is expected that these values are lower that the detection limit

# 5 Table la

Composition of the bottoms fraction of a fuels	
hydrocracker used in Examples 1 and 2	Mass %
Paraffinic compounds	68%
Naphthenic compounds	29%
Aromatic compounds	3 %

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# Comparative Experiment A and B

Example 1 was repeated with a 100% pure Fischer-Tropsch Waxy Raffinate feed of Table 1. The reactor temperature was varied to obtain a total liquid product (i.e. the total liquid effluent of the dewaxing step) having a pour point as in Example 1 or close to said pour point. See Table 2 for results and table 5 for composition.

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Example		A	2	B
Рееd	20/80	Fischer-	20/80	Fischer-
	blend	Tropsch WR	blend	Tropsch WR
Reactor temperature	304	315	297	307
(°C)				
Yield Base oil	43.2	37.3	45.2	34.9
between 400 and 460				
°C on feed (wt%)				
Base oil pour point	-31	-35	-17	-19
(ລູດ)				
Kinematic viscosity	4.15	4.022	4.109	3.879
at 100 °C of base oil			-	
(cSt)				
Viscosity Index of	129	130	132	133
base oil				

Table<sub>2</sub>

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### Example 3

12.7 weight parts of a wide boiling dewaxed and hydrofinished oil having the properties as listed in Table 3 was mixed with 87.3 weight parts of a Shell MDS waxy Raffinate having the properties listed in Table 3. The wide boiling dewaxed and hydrofinished oil was prepared by catalytic dewaxing of a bottoms fraction of a fuels hydrocracker followed by a hydrofinising step on the dewaxed effluent. The hydrocracker in turn was fed by a vacuum distillate of a mineral crude feed.

This blend of API Group II base oils was mixed with a Shell MDS waxy Raffinate having the properties listed in Table 3.

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Table 3

Table 3		
Feed	Wide	Shell
	boiling	MDS Waxy
	Mineral	Raffinat
	dewaxed	е
	and	
	hydrofini	
	shed Oil	
Density		
(D20/4)	0.847	0.784
Pour		
point		
(°C)	-24	45
Nitrogen		
(ppmw)	< 1	< 1
Sulphur		
(wmqq)	8	< 2
Kinematic		
viscosity		
at 100 °C	4.679	5.098

boiling		
point		
(IBP) °C		
IBP-390		
°C (wt%)	20.6	2.3
390-520		
°C (wt%)	67.9	88.4
520 °C -		
FBP (wt%)	11.5	9.3
Final		
boiling		
point		
(FBP) °C	600	573
Wax		•
content(w		
t%)	No wax	27.1

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The above blend was contacted with a dewaxing catalyst consisting of 0.7 wt% platinum, 25 wt% ZSM-12 and a silica binder. The dewaxing conditions were 40 bar hydrogen, WHSV = 1 kg/l.h, and a hydrogen gas rate of 700 Nl/kg feed. The experiment was carried out at two different reaction temperatures, namely 317 °C.

From the effluent of the dewaxing step a base oil boiling between 400 and 470 °C was isolated from lower and higher boiling products. The yield of this fraction was 40 wt% on the blended feed. The pour point was -30 °C, the Kinematic viscosity at 100 °C was 4,059 cSt and the Viscosity Index was 129. The composition of this fraction was analysed using the following technique.

The cyclo-paraffin (naphthenic compounds) content in this mixture of cyclo-, normal and iso-paraffins is

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measured by the following method. Any other method resulting in the same results may also be used. The base oil sample is first separated into a polar (aromatic) phase and a non-polar (saturates) phase by making use of a high performance liquid chromatography (HPLC) method IP368/01, wherein as mobile phase pentane is used instead of hexane as the method states. The saturates and aromatic fractions are then analyzed using a Finnigan MAT90 mass spectrometer equipped with a Field desorption/Field Ionisation (FD/FI) interface, wherein FI (a "soft" ionisation technique) is used for the quantitative determination of hydrocarbon types in terms of carbon number and hydrogen deficiency of this particular base oil fraction. The instrument conditions to achieve such a soft ionization technique are a source temperature of 30 °C, an extraction voltage of 5kV, an emitter current of 5mA and a probe temperature ramp of 40°C to 400°C (20°C/min)

The type classification of compounds in mass spectrometry is determined by the characteristic ions formed and is normally classified by "z number". This is given by the general formula for all hydrocarbon species: CnH2n+z. Because the saturates phase is analysed separately from the aromatic phase it is possible to determine the content of the different (cyclo)-paraffins having the same stoichiometry. The results of the mass spectrometer are processed using commercial software (poly 32; available from Sierra Analytics LLC, 3453 Dragoo Park Drive, Modesto, Calif. GA95350 USA) to determine the relative proportions of each hydrocarbon type and the average molecular weight and polydispersity of the saturates and aromatics fractions.

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For the base oil fraction boiling between 400 and 470  $^{\circ}\text{C}$  as obtained above the composition as listed in Table 5 was determined using the above technique.

Base oil of	Experiment B		888	118	1.8
Base oil of	Experiment A		%98	13%	t⊷I 9%
Base oil of	Example 3		83	16%	de □
Base Oil of	Example 2		828	178	&P □
Base Oil of	Example 1		748	25%	t} 910
Composition as	determined by	method example 3	Iso- and normal paraffins (wt%)	Naphthenic compounds (wt%)	Polars (wt%)

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#### CLAIMS

- 1. Process to prepare a base oil having an paraffin content of between 75 and 95 wt% by subjecting a mixture of a Fischer-Tropsch derived feed and a petroleum derived feed to a catalytic pour point reducing treatment.
- 2. Process according to claim 1, wherein the petroleum derived feed is a bottoms fraction of a fuels hydrocracker.

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- 3. Process according to claim 2, wherein the content of sulphur in the mixed feed to the pour point reducing treatment is below 50 ppm and the content of nitrogen in the mixed feed to the pour point reducing treatment is below 10 ppm.
- 4. Process according to any one of claims 1-3, wherein the wax content in the petroleum derived feed is below 50wt%, preferably below 30 wt%.
- 5. Process according to claim 4, wherein the pour point of the petroleum derived feed is below -10 °C.
- 6. Process according to any one of claims 1-5, wherein the petroleum derived feed has an aromatic content of between 0 and 20 wt% and a naphthenic compound content of preferably between 15 and 90 wt%.
- 7. Process according to claim 6, wherein the petroleum derived feed has a saturates content of greater than 98 wt% a viscosity index of between 80 and 150 and a sulphur content of below 0.001 wt%.
- 8. Process according to claim 7, wherein the petroleum derived feed has been obtained in a process involving a hydrofinishing step performed at a hydrogen pressure of greater than 100 bars.

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9. Process according to any one of claims 1-8, wherein the fraction of petroleum derived feed in the mixture is higher than 5 wt% and lower than 50 wt%.

- 10. Process according to any one of claims 1-9, wherein the base oil is hydrogenated after performing the pour point reducing treatment such that the content of aromatics is below 1 wt%.
- 11. Process according to any one of claims 1-10, wherein the catalytic pour point reducing treatment is a
- catalytic dewaxing process performed in the presence of a catalyst comprising a Group VIII metal and an intermediate pore size zeolite having pore diameter between 0.35 and 0.8 nm, and a binder.
- 12. Process according to any one of claims 1-11, wherein after performing the catalytic pour point reducing treatment hydrogen is separated from the dewaxed effluent, contacted with a heterogeneous adsorbent selective for removing hydrogen sulphide and recycled to said catalytic pour point reducing treatment.
- 13. Process according to claim 12, wherein the heterogeneous adsorbent is zinc oxide.
  - 14. Process according to any one of claims 1-13, wherein the Fischer-Tropsch feed is obtained by hydroisomerisation of a Fischer-Tropsch product.
- 15. Process according to claim 14, wherein the hydroisomerised Fischer-Tropsch feed is obtained by means of the following steps:
  - (a) hydrocracking/hydroisomerisating a Fischer-Tropsch product,
- (b) separating by means of distillation the product of step (a) into one or more gas oil fractions and a higher boiling Fischer-Tropsch derived feed.
  - 16. Process according to claim 15, wherein the Fischer-Tropsch product used as feed in step (a) is a product wherein the weight ratio of compounds having at least 60

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or more carbon atoms and compounds having at least 30 carbon atoms in the Fischer-Tropsch product is at least 0.4 and wherein at least 30 wt% of compounds in the Fischer-Tropsch product have at least 30 carbon atoms.

# INTERNATIONAL SEARCH REPORT

International Application No PCT/EP2004/051248

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invention. Advantageously the catalytic dewaxing will then not have to be performed at the higher pressure required for the subsequent hydrofinishing. In contrast it can be performed at a more preferred lower hydrogen pressure range of between 40 and 70 bars. Omitting such a hydrofinishing step is especially possible if the mineral derived dewaxed oils themselves are prepared by means of a process which does include a hydrofinishing step, preferably performed at a hydrogen pressure of above 100 bars. Examples of such hydrofinishing processes are for example those described below.

The oils are preferably made starting from a vacuum distillate or a de-asphalted vacuum residue of a mineral crude oil feedstock or from a waxy feed such as a slack wax, wherein the process includes a hydroprocessing step in which the sulphur and polar compounds are reduced to the preferred ranges. The viscosity index is preferably between 80-150, while good results have been achieved with oils having a viscosity index of between 80 and 120.

Preferably the T10wt% recovery point of this oil is between 200 and 450 °C, more preferably between 300 and 420 °C and the T90wt% recovery point is between 300 and 550 °C, more preferably between 400 and 550 °C.

By using such a wide boiling oil it has been found possible to reduce the iso-paraffin content of the resultant base oils for both the lower viscosity grades, ranging from 2 (cSt) at 100 °C kinematic viscosity, to and including the higher viscosity grades having a kinematic viscosity at 100 °C of 15 (cSt).

The dewaxed oil can be obtained as such by well known processes as for example described in Chapter 6 of Lubricant Base Oil and Wax Processing, Avilino Sequeira, Jr, Marcel Dekker, Inc. New York, 1994, page 119-150. The preferred wide boiling oil can be prepared by mixing various viscosity grades of, preferably API Group II or

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between 1.2-2 bara, wherein the gas oil product and lower boiling fractions, such as naphtha and kerosine fractions, are separated from the higher boiling fraction of the product of step (a). The higher boiling fraction, of which suitably at least 95 wt% boils above 370 °C, may be further separated in a vacuum distillation step wherein a higher boiling fraction is separated. The vacuum distillation is suitably performed at a pressure of between 0.001 and 0.05 bara.

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The vacuum distillation of step (b) is preferably operated such that the desired Fischer Tropsch derived feed is obtained boiling in the specified range and having a kinematic viscosity, which relates to the base oil end product(s) specification. The kinematic viscosity at 100 °C of the Fischer Tropsch derived feed is preferably between 3 and 10 cSt.

The mixture of petroleum derived and Fischer-Tropsch derived feeds will suitably have a viscosity corresponding to the desired viscosity of the base oil product. Preferably the kinematic viscosity at 100 °C of the mixture is between 3 and 10 (cSt) Suitable distillate fractions have a T10wt% boiling point of between 200 and 450 °C, preferably between 300 and 420 °C and a T90wt% boiling point of between 300 and 550 °C, preferably between 400 and 550 °C. The fraction of petroleum derived feed in the mixture is preferably higher than 5 wt%, more preferably higher than 10 wt% and preferably lower than 50 wt% and more preferably below 30 wt% and even more preferably below 25 wt%. The actual content of petroleum-derived feed in the mixture will of course depend on the paraffin content of said feed. The mixture will preferably contain less than 50 ppm sulphur and/or less that 10 ppm nitrogen.

With the catalytic pour point reducing treatment is understood every process wherein the pour point of the

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Co/Pt/Al<sub>2</sub>O<sub>3</sub>; Co/Pd/SiO<sub>2</sub>; Co/Sn/Al<sub>2</sub>O<sub>3</sub>; Ni/Sn/SiO<sub>2</sub>; Zn/Al<sub>2</sub>O<sub>3</sub>, ZnO/SiO<sub>2</sub>, Co/ZnO; Mo/ZnO; Ni/ZnO; Co/Mo/ZnO; Ni/Mo/ZnO; Pt/ZnO; Pt/Pd/ZnO. The adsorbent may also be employed as a bulk metal oxide or as a bulk metal, including but not necessarily limited to, a finely divided skeleton metal, including Raney metals, ponderous metals, Rieke metals, and metal sponges. The temperature and pressure conditions during said contacting are preferably within the ranges specified for the catalytic pour point reduction.

From the effluent of the pour point reducing treatment and the optional hydrogenation treatment one or more base oil grades may be isolated by means of fractionation. Base oil products having kinematic viscosity at 100 °C of between 2 and 10 CSt, having a volatility of between 8 and 11% (according to CEC L40 T87) and a pour point of between -20 and -60 °C (according to ASTM D 97) may advantageously be obtained.

The content of paraffins is more preferably less than 90 wt% and more preferably higher than 80 wt%.

The above-described base oil can suitably find use as base oil for an Automatic Transmission Fluids (ATF), motor engine oils, electrical oils or transformer oils and refrigerator oils. lubricant formulations such as motor engine oils of the OW-x and 5W-x specification according to the SAE J-300 viscosity classification, wherein x is 20, 30, 40, 50 or 60 may be advantageously made using this base oil.

It has been found that lubricant formulations can be prepared with the base oils obtainable by the process of the current invention without the need to add high contents of additional ester or aromatic co-base oils. Preferably less than 15 wt% and more preferably less than 10 wt% of such ester or aromatic co-base oil is present in such formulations.

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#### Example 1 and 2

A blend of 20 wt% of a bottoms fraction of a fuels hydrocracker and 80 wt% of a Fischer-Tropsch derived Waxy Raffinate as obtained from Shell MDS (Malaysia) Sdn Bhd (marketed as Shell MDS Waxy Raffinate) was prepared. The Shell MDS Waxy Raffinate is a partly isomerised Fischer-Tropsch derived fraction.

The above blend was contacted with a dewaxing catalyst consisting of 0.7 wt% platinum, 25 wt% ZSM-12 and a silica binder. The dewaxing conditions were 40 bar hydrogen, WHSV = 1 kg/l.h, and a hydrogen gas rate of 700 Nl/kg feed. The experiment was carried out at two different reaction temperatures, namely 304 °C(Example 1) and 297 °C (Example 2).

From the effluent of the dewaxing step a base oil boiling between 400 and 460 °C was isolated from lower and higher boiling products. This fraction was analysed. The yield and properties of this fraction having a kinematic viscosity of about 4 cSt are reported in Table 2. Table 8 lists the composition of these base oils.

Table 1

Feed	bottoms fraction of a fuels hydrocracker	Shell MDS Waxy Raffinate
Density (D70/4)	0.8036	0.7845
Pour point°C	+39	+42°C
Nitrogen (ppmw)	< 1	n.a. (*)
Sulphur (ppmw)	< 2	n.a.(*)

	<u></u>	
Kinematic		
viscosity	4.439	5.062
at 100 °C	4.439	3.002
Mm <sup>2</sup> /S (CSt)		
Initial		
boiling	269	361
point	209	301
(IBP) °C		
IBP-390	26.9	4.4
°C (wt%)	20.9	
390-520	63.0	84.8
°C (wt%)		
520 °C -	10.1	10.8
FBP (wt%)		
Final		
boiling	602	587
point	) 002	
(FBP) °C		
Wax		16.4
content	17.8	
(wt%)	·	
	and In wiou of	Fischer-Tronsch

\* N.a. = not analysed. In view of Fischer-Tropsch source it is expected that these values are lower that the detection limit

# 5 Table la

Composition of the bottoms	
fraction of a fuels	
hydrocracker used in Examples 1	
and 2	Mass %
Paraffinic compounds	68%
Naphthenic compounds	29%
Aromatic compounds	3 %

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# Comparative Experiment A and B

Example 1 was repeated with a 100% pure Fischer-Tropsch Waxy Raffinate feed of Table 1. The reactor temperature was varied to obtain a total liquid product (i.e. the total liquid effluent of the dewaxing step) having a pour point as in Example 1 or close to said pour point. See Table 2 for results and table 7 for composition.

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Example	1	A	2	B
Feed	20/80	Fischer-	20/80	Fischer-
	blend	Tropsch WR	blend	Tropsch WR
Reactor temperature	304	315	297	307
(°C)				
Yield Base oil	43.2	37.3	45.2	34.9
between 400 and 460		•		
°C on feed (wt%)				
Base oil pour point	-31	-35	-17	-19
(°C)				
Kinematic viscosity	4.15	4.022	4.109	3.879
at 100 °C of base oil				
Viscosity Index of	129	130	132	133
base oil		•		

[ab]e

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boiling		
point		
(IBP) °C		·
IBP-390		
°C (wt%)	20.6	2.3
390-520		
°C (wt%)	67.9	88.4
520 °C -		
FBP (wt%)	11.5	9.3
Final		
boiling		
point		
(FBP) °C	600	573
Wax		
content(w		
<b>t</b> %)	No wax	27.1

The above blend was contacted with a dewaxing catalyst consisting of 0.7 wt% platinum, 25 wt% ZSM-12 and a silica binder. The dewaxing conditions were 40 bar hydrogen, WHSV = 1 kg/l.h, and a hydrogen gas rate of 700 N1/kg feed. The experiment was carried out at two-different reaction temperatures, namely 317 °C.

boiling between 400 and 470 °C was isolated from lower and higher boiling products. The yield of this fraction was 40 wt% on the blended feed. The pour point was -30 °C, the Kinematic viscosity at 100 °C was 4,059 (cSt) and the Viscosity Index was 129. The composition of this fraction was analysed using the following technique.

The cyclo-paraffin (naphthenic compounds) content in this mixture of cyclo-, normal and iso-paraffins is

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For the base oil fraction boiling between 400 and 470 °C as obtained above the composition as listed in Table 8 was determined using the above technique.

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Composition as	Base Oil of	Base Oil of	Base oil of	Base oil of	Base old of
determined by	Example 1	Example 2	Example 3	Experiment A	Experiment B
method example 3					
Iso- and normal paraffins (wt%)	748	828	83	<del>8</del> 98	888
Naphthenic compounds (wt%)	25%	178	16%	138	118
Polars (wt%)	1.8°	1.8	de □	1.8	dip H

# PATENT COOPERATION TREATY

# PCT

# INTERNATIONAL PRELIMINARY REPORT ON PATENTABILITY

(Chapter II of the Patent Cooperation Treaty)

(PCT Article 36 and Rule 70) 1 2 OCT 2005 REC'D PCT WIPO Applicant's or agent's file reference FOR FURTHER ACTION **TS 5586 PCT** See Form PCT/IPEA/416 International application No. International filing date (day/month/year) Priority date (day/month/year) PCT/EP2004/051248 25.06.2004 27.06.2003 International Patent Classification (IPC) or national classification and IPC C10G45/58, C10G65/12 Applicant SHELL INTERNATIONALE RESEARCH MAATSCHAPPIJ B.V. This repart is the international preliminary examination report, established by this International Preliminary Examining Authority under Article 35 and transmitted to the applicant according to Article 36. This REPORT consists of a total of 5 sheets, including this cover sheet. This report is also accompanied by ANNEXES, comprising: a. A sent to the applicant and to the International Bureau) a total of 5 sheets, as follows: sheets of the description, claims and/or drawings which have been amended and are the basis of this report and/or sheets containing rectifications authorized by this Authority (see Rule 70.16 and Section 607 of the Administrative Instructions). sheets which supersede earlier sheets, but which this Authority considers contain an amendment that goes beyond the disclosure in the International application as filed, as indicated in Item 4 of Box No. I and the Supplemental Box. b. [ (sent to the International Bureau only) a total of (indicate type and number of electronic carrier(s)), containing a sequence listing and/or tables related thereto, in computer readable form only, as indicated in the Supplemental Box Relating to Sequence Listing (see Section 802 of the Administrative Instructions). This report contains indications relating to the following items: Box No. 1 Basis of the opinion Box No. II **Priority** Box No. III Non-establishment of opinion with regard to novelty, inventive step and industrial applicability Box No. IV Lack of unity of invention Box No. V Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement Certain documents cited Box No. VI ☐ Box No. VII Certain defects in the international application ☐ Box No. VIII Certain observations on the international application Date of submission of the demand Date of completion of this report 17.03.2005 11.10.2005 Name and malling address of the international preliminary examining authority: **Authorized Officer** European Patent Office - P.B. 5818 Patentlaan 2 NL-2280 HV Rijswijk - Pays Bas Deurinck, P Tel. +31 70 340 - 2040 Tx: 31 651 epo nl Fax: +31 70 340 - 3016 Telephone No. +31 70 340-2404

# INTERNATIONAL PRELIMINARY REPORT ON PATENTABILITY

International application No. PCT/EP2004/051248

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_	B0	x No. I	Basis of the report
1.	. Wi file	th regard d, unless	I to the language, this report is based on the international application in the language in which it was so therwise indicated under this item.
		inte	port is based on translations from the original language into the following language, s the language of a translation furnished for the purposes of: mational search (under Rules 12.3 and 23.1(b)) lication of the international application (under Rule 12.4) rnational preliminary examination (under Rules 55.2 and/or 55.3)
2.	Hav	o Decii	to the <b>elements*</b> of the international application, this report is based on (replacement sheets which furnished to the receiving Office in response to an invitation under Article 14 are referred to in this riginally filed" and are not annexed to this report):
	Des	cription,	Pages
	1, 3	-30	as originally filed
	2, 2	<b>a</b> .	received on 27.05.2005 with letter of 27.05.2005
Claims, Numbers			
	1-13	3	received on 27.05.2005 with letter of 27.05.2005
		a seque	ence listing and/or any related table(s) - see Supplemental Box Relating to Sequence Listing
3.		The am	endments have resulted in the cancellation of:
			lescription, pages
			laims, Nos. Irawings, sheets/figs
		☐ the s	equence listing (specify):
		□ any t	able(s) related to sequence listing <i>(specify)</i> :
4.	Had	HOL DEBI	ort has been established as if (some of) the amendments annexed to this report and listed below made, since they have been considered to go beyond the disclosure as filed, as indicated in the all Box (Rule 70.2(c)).
			escription, pages
			laims, Nos. rawings, sheets/figs
		☐ the s	equence listing (specify):
		⊔ any ta	able(s) related to sequence listing (specify):
	*	If item	n 4 applies, some or all of these sheets may be marked "superseded."

# INTERNATIONAL PRELIMINARY REPORT ON PATENTABILITY

International application No. PCT/EP2004/051248

Box No. V Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

1. Statement

Novelty (N)

Yes: Claims

1-13

No: Claims

Inventive step (IS)

Yes: Claims

1-13

No: Claims

Industrial applicability (IA)

Yes: Claims
No: Claims

1-13

2. Citations and explanations (Rule 70.7):

see separate sheet

PCT/EP2004/051248

# Re Item V

Reasoned statement with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

The following document is additionally mentioned:

D6: US 2003/0088133

# 1. Novelty

1.1

The present application meets the criteria of Article 33(1) PCT, because the subject-matter of independent Claim 1 is new in the sense of Article 33(2) PCT.

None of the prior art documents discloses a process to prepare a base oil having a paraffin content of between 75 and 95 wt.% by subjecting a mixture of a hydroisomerised Fischer-Tropsch wax and a petroleum derived feed to a catalytic pour point reducing treatment, wherein the petroleum derived feed has an aromatic content of between 0 and 20 wt.% and a naphthenic compound content of between 15 and 90 wt.% and wherein the fraction of petroleum derived feed in the mixture is higher than 5 wt.% and lower than 50 wt.%.

1.2

Claims 2-13 are dependent on independent Claim 1. and as such also meet the requirements of the PCT with respect to novelty.

# 2. Inventive Step

2.1

The present application meets the criteria of Article 33(1) PCT, because the subject-matter of independent Claim 1 involves an inventive step in the sense of Article 33(3) PCT.

# INTERNATIONAL PRELIMINARY REPORT ON PATENTABILITY (SEPARATE SHEET)

International application No.

PCT/EP2004/051248

Document D6, which is considered to represent the most relevant state of the art, discloses a process to prepare a Fischer-Tropsch derived base oil having a reduced content of paraffins by subjecting a Fischer-Tropsch wax to a hydroisomerization dewaxing process and adding at least one lube base stock composed of alkylaromatics, alkylcycloparaffins, or mixtures thereof in an amount of from 1 wt.% to 50 wt.% after the dewaxing step. This lube base stock is made by a complex process (see D6: claim 1; Figure 1).

The subject-matter of Claim 1 differs from D6 in that a petroleum derived feed having an aromatic content of between 0 and 20 wt.% and a naphthenic compound content of between 15 and 90 wt.% is added before the dewaxing step (distinguishing feature).

The *technical effect* that is achieved with this distinguishing feature is prepare a Fischer-Tropsch derived base oil having a reduced content of paraffins by a more simple process (see letter of applicant dated 27.05.2005).

The problem to be solved by the present invention may be regarded as: "To provide a more simple process to prepare a Fischer-Tropsch derived base oil having a reduced content of paraffins".

Starting from the disclosure of D6 and intending to solve the above-mentioned problem, a person skilled in the art had no incentive from D1-D5 to add a petroleum derived feed having an aromatic content of between 0 and 20 wt.% and a naphthenic compound content of between 15 and 90 wt.% before the dewaxing step.

Thus the subject-matter of independent Claim 1 involves an inventive step.

## 2.2

Claims 2-13 are dependent on independent Claim 1. and as such also meet the requirements of the PCT with respect to inventive step.



Europäisches **Patentamt** 

European **Patent Office**  Office européen des brevets

> REC'D 07 SEP 2004 WIPO PCT

Bescheinigung

Certificate

Attestation

Die angehefteten Unterlagen stimmen mit der ursprünglich eingereichten Fassung der auf dem nächsten Blatt bezeichneten europäischen Patentanmeldung überein.

The attached documents are exact copies of the European patent application conformes à la version described on the following page, as originally filed.

Les documents fixés à cette attestation sont initialement déposée de la demande de brevet européen spécifiée à la page suivante.

Patent application No. Demande de brevet n° Patentanmeldung Nr.

03291598.5



Der Präsident des Europäischen Patentamts; Im Auftrag

For the President of the European Patent Office

Le Président de l'Office européen des brevets p.o.

R C van Dijk



Europäisches Patentamt European Patent Office Office européen des brevets

Anmeldung Nr:

Application no.:

03291598.5

Demande no:

Anmeldetag:

Date of filing: 27.06.03

Date de dépôt:

Anmelder/Applicant(s)/Demandeur(s):

SHELL INTERNATIONALE RESEARCH MAATSCHAPPIJ B.V. Carel van Bylandtlaan 30 2596 HR Den Haag PAYS-BAS

Bezeichnung der Erfindung/Title of the invention/Titre de l'invention: (Falls die Bezeichnung der Erfindung nicht angegeben ist, siehe Beschreibung. If no title is shown please refer to the description. Si aucun titre n'est indiqué se referer à la description.)

Process to prepare a lubricating base oil

In Anspruch genommene Prioriät(en) / Priority(ies) claimed /Priorité(s) revendiquée(s)
Staat/Tag/Aktenzeichen/State/Date/File no./Pays/Date/Numéro de dépôt:

Internationale Patentklassifikation/International Patent Classification/Classification internationale des brevets:

C10G/

Am Anmeldetag benannte Vertragstaaten/Contracting states designated at date of filing/Etats contractants désignées lors du dépôt:

AT BE BG CH CY CZ DE DK EE ES FI FR GB GR HU IE IT LU MC NL PT RO SE SI SK TR LI

TS 5586 EPC

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#### PROCESS TO PREPARE A LUBRICATING BASE OIL

The invention is directed to a process to prepare a base oil having an paraffin content of between 75 and 95 wt%.

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WO-A-0157166 describes the use of a highly paraffinic base oil as obtained from a Fischer-Tropsch wax in a motor engine lubricant formulation. The examples illustrate that such formulations will also consist of an ester, which according to the description of the patent are added to confer additional desired characteristics, such as additive solvency.

The use of ester co-base fluids in lubricant formulations as illustrated in WO-A-0157166 is not desired because such ester co-base fluids are not widely available and thus expensive. Additive solvency may be improved by using a paraffinic base stock, which contains less paraffins. Such base oils may be prepared by hydroisomerisation of petroleum derived waxes followed by a solvent or catalytic dewaxing step. A disadvantage of such a process is that the starting petroleum derived waxes, such as for example slack wax, are not easily obtainable. Furthermore such waxes may not always have the desired high paraffin content needed to make the desired base oils as per this invention.

The object of the present invention is to provide a process wherein a base oil with a paraffin content of between 75 and 95 wt% is obtained which does not have the disadvantages of the prior art processes.

This object is achieved by the following process. Process to prepare a base oil having an paraffin content of between 75 and 95 wt% by subjecting a mixture of a

Fischer-Tropsch derived feed and a petroleum derived feed to a catalytic pour point reducing treatment.

Applicants found that by mixing a relatively small amount of a petroleum derived feed with a Fischer-Tropsch derived feed before performing a catalytic pour point reducing treatment a base oil may be obtained having the desired properties.

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The petroleum-derived fraction may in principle be any fraction boiling in the base oil range and containing non-paraffinic compounds. Preferably a petroleum-derived fraction is used which has been subjected to a hydroprocessing step in order to reduce aromatic, sulphur and nitrogen content of such fractions and improve some of the desired properties such viscosity index. The hydroprocessing step may be a hydrotreating optionally followed by a hydrocracking step. Such processes are for example performed when preparing base oils from a petroleum derived vacuum distillate or de-asphalted oils.

A very interesting petroleum derived feed is the bottoms fraction of a fuels hydrocracker. With a fuels hydrocracker in the context of the present invention is meant a hydrocracker process which main products are naphtha, kerosene and gas oil. The conversion, expressed in the weight percentage of the fraction in the feed to the hydrotreater-hydrocracker which boils above 370 °C which are converted to products boiling below 370 °C, in the hydrotreater-hydrocracker process is typically above 50 wt%. Examples of possible fuels hydrocracker processes, which may yield a bottoms fraction which can be used in the present process, are described in the above referred to EP-A-699225, EP-A-649896, WO-A-9718278, EP-A-705321, EP-A-994173 and US-A-4851109.

Another interesting petroleum derived feed is the fraction obtained in a dedicated base oil hydrotreater-hydrocracker. In such a hydrotreater-hydrocracker the

main products will boil in the base oil range. Typically such processes operate at a feed conversion of below 50 wt% and more typically between 20 and 40 wt%. The petroleum derived feed is thus the high boiling fraction as obtained in such a process prior to dewaxing.

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Preferably the fuels hydrocracker is operated in two steps, consisting of a preliminary hydrotreating step followed by a hydrocracking step. In the hydrotreating step nitrogen and sulphur are removed and aromatics are saturated to naphthenes

The Fischer-Tropsch derived feed preferably is a hydroisomerized Fischer-Tropsch wax. Such a feed may be obtained by well-known processes, for example the so-called commercial Sasol process, the Shell Middle Distillate Process or by the non-commercial Exxon process. These and other processes are for example described in more detail in EP-A-776959, EP-A-668342, US-A-4943672, US-A-5059299, WO-A-9934917 and WO-A-9920720. The process will generally comprise a Fischer-Tropsch synthesis and a hydroisomerisation step as described in these publications.

The mixture of petroleum derived and Fischer-Tropsch derived feeds will suitably have a viscosity corresponding to the desired viscosity of the base oil product. Preferably the kinematic viscosity at 100 °C of the mixture is between 3 and 10 cSt. Suitable distillate fractions obtained in step (a) have a TlOwt% boiling point of between 200 and 450 °C and a T90wt% boiling point of between 300 and 550 °C. The fraction of petroleum derived feed in the mixture is preferably higher than 5 wt%, more preferably higher than 10 wt% and preferably lower than 50 wt% and more preferably below 30 wt% and even more preferably below 25 wt%. The actual content of petroleum-derived feed in the mixture will of course depend on the paraffin content of said feed. The

mixture will preferably contain less than 50 ppm sulphur and/or less that 10 ppm nitrogen.

With the catalytic pour point reducing treatment is understood every process wherein the pour point of the base oil is reduced by more than 10 °C, preferably more than 20 °C, more preferably more than 25 °C.

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The catalytic dewaxing or pour point reducing process can be performed by any process wherein in the presence of a catalyst and hydrogen the pour point of the base oil precursor fraction is reduced as specified above. Suitable dewaxing catalysts are heterogeneous catalysts comprising a molecular sieve and optionally in combination with a metal having a hydrogenation function, such as the Group VIII metals. Molecular sieves, and more suitably intermediate pore size zeolites, have shown a good catalytic ability to reduce the pour point of the distillate base oil precursor fraction under catalytic dewaxing conditions. Preferably the intermediate pore size zeolites have a pore diameter of between 0.35 and 0.8 nm. Suitable intermediate pore size zeolites are mordenite, ZSM-5, ZSM-12, ZSM-22, ZSM-23, SSZ-32, ZSM-35 and ZSM-48. Another preferred group of molecular sieves are the silica-aluminaphosphate (SAPO) materials of which SAPO-11 is most preferred as for example described in US-A-4859311. ZSM-5 may optionally be used in its HZSM-5 form in the absence of any Group VIII metal. The other molecular sieves are preferably used in combination with an added Group VIII metal. Suitable Group VIII metals are nickel, cobalt, platinum and palladium. Examples of possible combinations are Ni/ZSM-5, Pt/ZSM-23, Pd/ZSM-23, Pt/ZSM-48 and Pt/SAPO-11. Further details and examples of suitable molecular sieves and dewaxing conditions are for example described in WO-A-9718278, US-A-5053373, US-A-5252527 and US-A-4574043.

The dewaxing catalyst suitably also comprises a binder. The binder can be a synthetic or naturally occurring (inorganic) substance, for example clay, silica and/or metal oxides. Natural occurring clays are for example of the montmorillonite and kaolin families. The binder is preferably a porous binder material, for example a refractory oxide of which examples are: alumina, silica-alumina, silica-magnesia, silicazirconia, silica-thoria, silica-beryllia, silica-titania as well as ternary compositions for example silicaalumina-thoria, silica-alumina-zirconia, silica-aluminamagnesia and silica-magnesia-zirconia. More preferably a low acidity refractory oxide binder material which is essentially free of alumina is used. Examples of these binder materials are silica, zirconia, titanium dioxide, germanium dioxide, boria and mixtures of two or more of these of which examples are listed above. The most preferred binder is silica.

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A preferred class of dewaxing catalysts comprise intermediate zeolite crystallites as described above and a low acidity refractory oxide binder material which is essentially free of alumina as described above, wherein the surface of the aluminosilicate zeolite crystallites has been modified by subjecting the aluminosilicate zeolite crystallites to a surface dealumination treatment. A preferred dealumination treatment is by contacting an extrudate of the binder and the zeolite with an aqueous solution of a fluorosilicate salt as described in for example US-A-5157191 or WO-A-2000029511. Examples of suitable dewaxing catalysts as described above are silica bound and dealuminated Pt/ZSM-5, silica bound and dealuminated Pt/ZSM-23, silica bound and dealuminated Pt/ZSM-12, silica bound and dealuminated Pt/ZSM-22 as for example described in WO-A-200029511 and EP-B-832171.

Catalytic dewaxing conditions are known in the art and typically involve operating temperatures in the range of from 200 to 500 °C, suitably from 250 to 400 °C, hydrogen pressures in the range of from 10 to 200 bar, preferably from 40 to 70 bar, weight hourly space velocities (WHSV) in the range of from 0.1 to 10 kg of oil per litre of catalyst per hour (kg/1/hr), suitably from 0.2 to 5 kg/1/hr, more suitably from 0.5 to 3 kg/1/hr and hydrogen to oil ratios in the range of from 100 to 2,000 litres of hydrogen per litre of oil. By varying the temperature between 315 and 375 °C at between 40-70 bars, in the catalytic dewaxing step it is possible to prepare base oils having different pour point specifications varying from suitably lower than -60 to -10 °C.

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After performing the pour point reducing treatment lower boiling compounds formed during said treatment are suitably removed, preferably by means of distillation, optionally in combination with an initial flashing step.

The effluent of the pour point reducing treatment may suitably be subjected to a hydrogenation treatment. Hydrogenation may be performed on the entire effluent or on specific base oil grades after the above described fractionation. This may be required in order to reduce the content of aromatic compounds in the reduced pour point product to preferably values of below 1 wt%. Such a hydrogenation is also referred to as a hydrofinishing step. This step is suitably carried out at a temperature between 180 and 380 °C, a total pressure of between 10 to 250 bar and preferably above 100 bar and more preferably between 120 and 250 bar. The WHSV (Weight hourly space velocity) ranges from 0.3 to 2 kg of oil per litre of catalyst per hour (kg/l.h). Preferably a hydrogenation is performed in the same reactor as the catalytic dewaxing reactor. In such a reactor the beds of dewaxing catalyst

and hydrogenation catalyst will be placed in a stacked bed on top of each other.

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The hydrogenation catalyst is suitably a supported catalyst comprising a dispersed Group VIII metal. Possible Group VIII metals are cobalt, nickel, palladium and platinum. Cobalt and nickel containing catalysts may also comprise a Group VIB metal, suitably molybdenum and tungsten. Suitable carrier or support materials are low acidity amorphous refractory oxides. Examples of suitable amorphous refractory oxides include inorganic oxides, such as alumina, silica, titania, zirconia, boria, silica-alumina, fluorided alumina, fluorided silica-alumina and mixtures of two or more of these.

Examples of suitable hydrogenation catalysts are nickel-molybdenum containing catalyst such as KF-847 and KF-8010 (AKZO Nobel) M-8-24 and M-8-25 (BASF), and C-424, DN-190, HDS-3 and HDS-4 (Criterion); nickel-tungsten containing catalysts such as NI-4342 and NI-4352 (Engelhard) and C-454 (Criterion); cobalt-molybdenum containing catalysts such as KF-330 (AKZO-Nobel), HDS-22 (Criterion) and HPC-601 (Engelhard). Preferably platinum containing and more preferably platinum and palladium containing catalysts are used. Preferred supports for these palladium and/or platinum containing catalysts are amorphous silica-alumina. Examples of suitable silicaalumina carriers are disclosed in WO-A-9410263. A preferred catalyst comprises an alloy of palladium and platinum preferably supported on an amorphous silicaalumina carrier of which the commercially available catalyst C-624 of Criterion Catalyst Company (Houston, TX) is an example.

From the effluent of the pour point reducing treatment and the optional hydrogenation treatment one or more base oil grades may be isolated by means of fractionation. Base oil products having kinematic

viscosity at 100 °C of between 2 and 10 cSt, having a volatility of between 8 and 11% (according to CEC L40 T87) and a pour point of between -20 and -60 °C (according to ASTM D 97) may advantageously be obtained.

The content of paraffins is more preferably less than 90 wt% and more preferably higher than 80 wt%.

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The above-described base oil can suitably find use as base oil for an Automatic Transmission Fluids (ATF), motor engine oils, electrical oils or transformer oils and refrigerator oils. lubricant formulations such as motor engine oils of the OW-x and 5W-x specification according to the SAE J-300 viscosity classification, wherein x is 20, 30, 40, 50 or 60 may be advantageously made using this base oil.

It has been found that lubricant formulations can be prepared with the base oils obtainable by the process of the current invention without the need to add high contents of additional ester or aromatic co-base oils. Preferably less than 15 wt% and more preferably less than 10 wt% of such ester or aromatic co-base oil is present in such formulations.

## CLAIMS

- 1. Process to prepare a base oil having an paraffin content of between 75 and 95 wt% by subjecting a mixture of a Fischer-Tropsch derived feed and a petroleum derived feed to a catalytic pour point reducing treatment.
- 2. Process according to claim 1, wherein the petroleum derived feed is a bottoms fraction of a fuels hydrocracker.
  - 3. Process according to claim 2, wherein the content of sulphur in the mixed feed to the pour point reducing treatment is below 50 ppm.
  - 4. Process according to any one of claims 2-3, wherein the content of nitrogen in the mixed feed to the pour point reducing treatment is below 10 ppm.
  - 5. Process according to any one of claims 1-4, wherein the base oil is hydrogenated after performing the pour point reducing treatment such that the content of aromatics is below 1 wt%.
- 6. Process according to any one of claims 1-5, wherein the catalytic pour point reducing treatment is a catalytic dewaxing process performed in the presence of a catalyst comprising a Group VIII metal and an intermediate pore size zeolite having pore diameter between 0.35 and 0.8 nm, and a binder.
  - 7. Lubricant composition comprising a base oil as obtained from the process according to any one of claims 1-6 and less than 15 wt% of an ester or aromatic co-base oil.
    - 8. Lubricant according to claim 7, wherein the content of the ester or aromatic co-base oil is less than 10 wt%.

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TS 5586 EPC

# ABSTRACT

PROCESS TO PREPARE A LUBRICATING BASE OIL

Process to prepare a base oil having an paraffin content of between 75 and 95 wt% by subjecting a mixture of a Fischer-Tropsch derived feed and a petroleum derived feed to a catalytic pour point reducing treatment.

PCT/EP2004/051248



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Shell International B.V.

PO Box 384

Intellectual Property Services

3 November 2006

Our ref:

TS 5586 EPC P

European patent application No. 04 766 080.8-1213 Shell Internationale Research Maatschappij B.V.

#### Dear Sir or Madam

Please find our response to the communication pursuant Art 96(2) EPC regarding the above referred to application.

#### **Amendments**

We herewith submit an amended set of claims. Claims 2 and 7 have been deleted. Claims 3 to 13 have been renumbered accordingly. Claim 12 has been amended. Basis for this amendment can be found on page3, lines 24 to 27, and page 14 lines 25 to 28. Claim 12 has been renumbered accordingly as a new Claim 10.

We respectfully request the following correction under the Rule 88 EPC:

- -Page 22, line 19, page 24, line 9, page 29, line 3, and page 30, table heading, wherein the term "Table 5" should be replaced by the term "Table 4". For your consideration these pages are submitted as hand-amended pages.
- -Page 27, line 7, the sentence "The experiment was carried out at two different reaction temperatures, namely 317 °C" should read "The experiment was carried out at reaction temperature 317 °C". For the person skilled in art it would be evident that nothing else would have been intended than what is offered as the correction, since the remaining description of the experiment only relates to one reaction temperature. For your consideration this page is submitted as hand-amended page.

Furthermore we submit hand-amended pages 5, 14, 20, 23, 25, 27 where, as requested by the examiner, the unit "cSt" has been replaced by the term "mm²/s (cSt)", with the present expression retained in the parentheses after the appropriate SI unit (Guidelines, C-II, Annex 1).

Established at The Hague: Carel van Bylandslaan 30 Commercial Register, The Hague 27 155369 VAT number: NIOO4790996B58

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European Patent Office

03.11.2006

München

# Clarity

The examiner states that Claim 1 attempts to define the subject matter in terms of the result to be achieved (a base oil having a paraffin content of between 75 and 95 wt. %) and that the technical features to obtain this result should be clearly stated.

The applicant is of the opinion that 75 – 95 wt.% feature is not an inherent feature of the obtained product of the claimed process. The paraffin content of the resulting base oil will depend on the composition of the petroleum derived feed and the fraction of said feed in the total feed to be subjected to the pour point reducing treatment. It is possible for a skilled person to choose a suitable fraction of the petroleum derived feed to arrive at the claimed composition without undue experimentation.

We note that no objection has been raised versus novelty or inventive step. We furthermore trust the above information overcomes the objections raised versus clarity.

Yours faithfully

Shell International B.V.

M Cramwinckel (GA 17132)

Patent Attorney

(Ziv)

Enclosure:

New set of claims

Hand-amended pages 5, 14, 20, 22, 23, 24, 25, 27, and 30.

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03 11. 2006

TS 5586 EPC P



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## AMENDED CLAIMS

- 1. Process to prepare a base oil having a paraffin content of between 75 and 95 wt% by subjecting a mixture of a hydroisomerised Fischer-Tropsch wax and a petroleum derived feed to a catalytic pour point reducing treatment, wherein the petroleum derived feed has an aromatic content of between 0 and 20 wt% and a naphthenic compound content of between 15 and 90 wt% and wherein the fraction of petroleum derived feed in the mixture is higher than 5 wt% and lower than 50 wt%.
- 2. Process according to claim 1, wherein the content of sulphur in the mixed feed to the pour point reducing treatment is below 50 ppm and the content of nitrogen in the mixed feed to the pour point reducing treatment is below 10 ppm.
- 3. Process according to any one of claims 1-2, wherein the wax content in the petroleum derived feed is below 30 wt%.
  - 4. Process according to claim 3, wherein the pour point of the petroleum derived feed is below -10 °C.
- 5. Process according to any one of the claims 1-4, wherein the petroleum derived feed has a saturates content of greater than 98 wt% a viscosity index of between 80 and 150 and a sulphur content of below 0.001 wt%.
- 6. Process according to any one of claims 1-5, wherein the base oil is hydrogenated after performing the pour point reducing treatment such that the content of aromatics is below 1 wt%.

- 7. Process according to any one of claims 1-6, wherein the catalytic pour point reducing treatment is a catalytic dewaxing process performed in the presence of a catalyst comprising a Group VIII metal and an
- intermediate pore size zeolite having pore diameter between 0.35 and 0.8 nm, and a binder.
  - 8. Process according to any one of claims 1-7, wherein after performing the catalytic pour point reducing treatment hydrogen is separated from the dewaxed
- effluent, contacted with a heterogeneous adsorbent selective for removing hydrogen sulphide and recycled to said catalytic pour point reducing treatment.
  - 9. Process according to claim 8, wherein the heterogeneous adsorbent is zink oxide.
- 10. Process to prepare a base oil having a paraffin content of between 75 and 95 wt % by subjecting a mixture of a hydroisomerised Fisher-Tropsch derived wax and a petroleum derived feed to a catalytic pour point reducing treatment, wherein:
- (i) the petroleum derived feed has an aromatic content of between 0 and 20 wt% and wherein the fraction of petroleum derived feed in the mixture is higher than 5 wt% and lower than 50 wt%,

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- (ii) the hydroisomerised Fisher-Tropsch derived wax is obtained by means of the following steps:
- (a) hydrocracking/hydroisomerisating a Fisher Tropsch product,
- (b) separating by means of distillation the product of step (a) into one or more gas oil fractions and the higher boiling Fisher-Tropsch wax.
- 11. Process according to claim 10, wherein the Fischer-Tropsch product used as feed in step (a) is a product wherein the weight ratio of compounds having at least 60

or more carbon atoms and compounds having at least 30 carbon atoms in the Fischer-Tropsch product is at least 0.4 and wherein at least 30 wt% of compounds in the Fischer-Tropsch product have at least 30 carbon atoms.

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P.B.5818 - Patentlaan 2 2280 HV Rijswijk (ZH) (070) 3 40 20 40 FAX (070) 3 40 30 16 Europäisches Patentamt European Patent Office Office européen des brevets

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**EPO Customer Services** 

Tel.: +31 (0)70 340 45 00

Date

07-09-2006

Reference TS 5586 EPC P Application No./Patent No.

04766080.8 - 1213 PCT/EP2004051248

Applicant/Proprietor

SHELL INTERNATIONALE RESEARCH MAATSCHAPPIJ B.V.

#### Communication pursuant to Rules 109 and 110 EPC

## (1) Amendment of application documents, especially the claims (R. 109 EPC)

The above mentioned international (Euro-PCT) application has entered the European phase, or can do so, once the necessary conditions are fulfilled.

Under Articles 28, 41 PCT, Rules 52, 78 PCT and Rule 86(2) to (4) EPC, the applicant may amend the application documents after receiving the international search report.

Whether or not he has already done so, he now has a further opportunity to file amended claims or other application documents within a non-extendable time limit of one month after notification of the present communication (R. 109 EPC).

The claims applicable on expiry of the above time limit, i.e. those filed on entry into the European phase or in response to the present communication, will form the basis for the calculation of any claims fee to be paid (see page 2) and for any supplementary search to be carried out under Article 157(2) EPC (R. 109 EPC).



#### (2) Claims fees under Rule 110 EPC

Date

If the application documents on which the European grant procedure is to be based comprise more than ten claims, a claims fee shall be payable for the eleventh and each subsequent claim within the period provided for in Rule 107(1) EPC.

M	Based on the application documents currently on file, all necessary claims fees have already been paid
	(or the documents do not comprise more than 10 claims).

All necessary fees will be/have been debited automatically according to the automatic debit order.

The claims fee due for the claims ...... to .......... were not paid within the above-mentioned period.

Any non-paid claims fee, either based on the current set of claims or on any amended claims to be filed pursuant to Rule 109 EPC (see page 1), may still be validly paid within a non-extendable period of grace of one month after notification of this communication.

If a payment is made for only some of the claims, it must be indicated for which claims it is intended. If a claims fee is not paid in due time, the claim concerned is deemed to be abandoned (R. 110(4) EPC).

If claims fees have already been paid, but on expiry of the above-mentioned time limit there is a new set of claims containing fewer fee-incurring claims than previously, the claims fees in excess of those due under Rule 110(2), 2nd sentence, EPC will be refunded (R. 110(3) EPC).

You are reminded that any supplementary search under Article 157(2) EPC will relate only to the last set of claims applicable on expiry of the above time limit AND will be confined to those fee-incurring claims for which fees have been paid in due time.

The fee for the eleventh and each subsequent claim is EUR 45,00.

**Receiving Section** 



Please ignore Epo form 1226 dated 09/05/2006

Ligtvoet, Jan



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L

**EPA/EPO/DEB**  $oxed{\Xi}$ D-80298 München

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SHELL INTERNATIONALE RESEARCH

Europäisches **Patentamt** 

European **Patent Office**  Office européen des brevets

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Branch at The Hague

Primary Examiner (substantive examination) +31 70 340-2404

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Application No. 04 766 080.8 - 1213 TS 5586 EPC P

Date

06.09.2006

**Applicant** 

MAATSCHAPPIJ B.V.

2501 CH Den Haag

P.O. Box 302

PAYS-BAS

Intellectual Property Division,

SHELL INTERNATIONALE RESEARCH MAATSCHAPPIJ B.V.

#### Communication pursuant to Article 96(2) EPC

The examination of the above-identified application has revealed that it does not meet the requirements of the European Patent Convention for the reasons enclosed herewith. If the deficiencies indicated are not rectified the application may be refused pursuant to Article 97(1) EPC.

You are invited to file your observations and insofar as the deficiencies are such as to be rectifiable, to correct the indicated deficiencies within a period

#### months of

from the notification of this communication, this period being computed in accordance with Rules 78(2) and 83(2) and (4) EPC.

One set of amendments to the description, claims and drawings is to be filed within the said period on separate sheets (Rule 36(1) EPC).

Failure to comply with this invitation in due time will result in the application being deemed to be withdrawn (Article 96(3) EPC).



Deurinck, P **Primary Examiner** for the Examining Division

Enclosure(s):

4 page/s reasons (Form 2906)



Bescheid/Protokoll (Anlage)

Communication/Minutes (Annex)

Notification/Procès-verbal (Annexe)

**Datum** Date Date

06.09.2006

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1

Anmelde-Nr.: Demande nº:

Application No.: 04 766 080.8

The examination is being carried out on the following application documents:

#### **Description, Pages**

1, 3-30

as originally filed

2, 2a

as annexed to the Int. Prel. Examination Report

#### Claims, Numbers

1-13

as annexed to the Int. Prel. Examination Report

The following document is referred to in this communication; the numbering will be adhered to in the rest of the procedure:

D6: US 2003/0088133

#### Art. 84 EPC 1.

The application does not meet the requirements of Article 84 EPC, because Claims 1, 2, 7 and 12 are not clear for the following reasons:

1.1

Claim 1 attempts to define the subject-matter in terms of the result to be achieved (a base oil having a paraffin content of between 75 and 95 wt.%). However, the technical features to obtain this result should be clearly stated. Should this property be an inherent property of the obtained product, then this part of Claim 1 is superfluous. However, if further technical features are necessary in order to achieve this property, this have to be included in Claim 1.

1.2

Claims 2, 7 and 12 are process Claims using a specific product ("a petroleum derived feed" and "Fischer-Tropsch feed"). Process Claims disclosing products defined in terms of a process of manufacture are admissible only if the process using the products as



Bescheid/Protokoli (Anlage)

Communication/Minutes (Annex)

Notification/Procès-verbal (Annexe)

**Datum** Date

Date

06.09.2006

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2

Anmelde-Nr.: Application No.: 04 766 080.8 Demande nº:

such fulfil the requirements of patentability, i.e. that they are new and inventive. A process using a specific product is not rendered novel merely by the fact that it is produced by means of a new process (see Guidelines C.III, 4.7b).

#### 2. Novelty (Art. 54 EPC)

#### 2.1

The present application meets the requirements of Article 52(1) EPC, because the subject-matter of independent Claim 1 is new in the sense of Article 54(1) and (2) EPC.

None of the prior art documents discloses a process to prepare a base oil having a paraffin content of between 75 and 95 wt.% by subjecting a mixture of a hydroisomerised Fischer-Tropsch wax and a petroleum derived feed to a catalytic pour point reducing treatment, wherein the petroleum derived feed has an aromatic content of between 0 and 20 wt.% and a naphthenic compound content of between 15 and 90 wt.% and wherein the fraction of petroleum derived feed in the mixture is higher than 5 wt.% and lower than 50 wt.%.

#### 2.2

Claims 2-13 are dependent on independent Claim 1 and as such also meet the requirements of the EPC with respect to novelty.

#### 3. Inventive steps (Art. 56 EPC)

#### 3.1

The present application meets the requirements of Article 52(1) EPC, because the subject-matter of independent Claim 1 involves an inventive step in the sense of Article 56 EPC.

Document D6, which is considered to represent the most relevant state of the art, discloses a process to prepare a Fischer-Tropsch derived base oil having a reduced content of paraffins by subjecting a Fischer-Tropsch wax to a hydroisomerization dewaxing process and adding at least one lube base stock composed of alkylaromatics,



Bescheid/Protokoll (Anlage)

Communication/Minutes (Annex)

Notification/Procès-verbal (Annexe)

Datum Date Date

06.09.2006

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3

Anmelde-Nr.: Demande nº:

Application No.: 04 766 080.8

alkylcycloparaffins, or mixtures thereof in an amount of from 1 wt.% to 50 wt.% after the dewaxing step. This lube base stock is made by a complex process (see D6: claim 1; Figure 1).

The subject-matter of Claim 1 differs from D6 in that a petroleum derived feed having an aromatic content of between 0 and 20 wt.% and a naphthenic compound content of between 15 and 90 wt.% is added before the dewaxing step (distinguishing feature).

The technical effect that is achieved with this distinguishing feature is prepare a Fischer-Tropsch derived base oil having a reduced content of paraffins by a more simple process (see letter of applicant dated 27.05.2005).

The problem to be solved by the present invention may be regarded as: "To provide a more simple process to prepare a Fischer-Tropsch derived base oil having a reduced content of paraffins".

Starting from the disclosure of D6 and intending to solve the above-mentioned problem, a person skilled in the art had no incentive from D1-D5 to add a petroleum derived feed having an aromatic content of between 0 and 20 wt.% and a naphthenic compound content of between 15 and 90 wt.% before the dewaxing step.

Thus the subject-matter of independent Claim 1 involves an inventive step.

3.2

Claims 2-13 are dependent on independent Claim 1 and as such also meet the requirements of the EPC with respect to inventive step.

#### Further remarks 4.

The unit "cSt" expressed throughout the description does not meet the requirements of Rule 35(12) EPC and should be replaced by the appropriate SI unit (cf. the Guidelines,



Bescheld/Protokoll (Anlage)

Communication/Minutes (Annex)

Notification/Procès-verbal (Annexe)

Datum Date Date

06.09.2006

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4

Anmelde-Nr.:
Application No.: 04 766 080.8
Demande n°:

C-II, Annex 1). The present expressions should, however, be retained in parentheses after the replacement expressions.

## 5. <u>Conclusion</u>

The applicant is requested to file amended Claims which take account of the above comments. When filing amended Claims the applicant should at the same time bring the description into conformity with the amended Claims. Care should be taken during revision, especially of the introductory portion and any statements of problem or advantage, not to add subject-matter which extends beyond the content of the application as originally filed (Article 123(2) EPC).



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Date

09-05-2006

Reference TS 5586 EPC P Application No./Patent No.

04766080.8 - 1213 PCT/EP2004051248

Applicant/Proprietor

SHELL INTERNATIONALE RESEARCH MAATSCHAPPIJ B.V.

#### Communication pursuant to Rules 109 and 110 EPC

#### (1) Amendment of application documents, especially the claims (R. 109 EPC)

The above mentioned international (Euro-PCT) application has entered the European phase, or can do so, once the necessary conditions are fulfilled.

Under Articles 28, 41 PCT, Rules 52, 78 PCT and Rule 86(2) to (4) EPC, the applicant may amend the application documents after receiving the international search report.

Whether or not he has already done so, he now has a further opportunity to file amended claims or other application documents within a non-extendable time limit of one month after notification of the present communication (R. 109 EPC).

The claims applicable on expiry of the above time limit, i.e. those filed on entry into the European phase or in response to the present communication, will form the basis for the calculation of any claims fee to be paid (see page 2) and for any supplementary search to be carried out under Article 157(2) EPC (R. 109 EPC).



#### (2) Claims fees under Rule 110 EPC

Date

If the application documents on which the European grant procedure is to be based comprise more than ten claims, a claims fee shall be payable for the eleventh and each subsequent claim within the period provided for in Rule 107(1) EPC.

Based on the application documents currently on file, all necessary claims fees have already been pai (or the documents do not comprise more than 10 claims).		
All necessary fees will be/have been debited automatically according to the automatic debit order.		

The claims fees due for the claims 11 to 0013 were not paid within the above-mentioned period.

Any non-paid claims fee, either based on the current set of claims or on any amended claims to be filed pursuant to Rule 109 EPC (see page 1), may still be validly paid within a non-extendable period of grace of one month after notification of this communication.

If a payment is made for only some of the claims, it must be indicated for which claims it is intended. If a claims fee is not paid in due time, the claim concerned is deemed to be abandoned (R. 110(4) EPC).

If claims fees have already been paid, but on expiry of the above-mentioned time limit there is a new set of claims containing fewer fee-incurring claims than previously, the claims fees in excess of those due under Rule 110(2), 2nd sentence, EPC will be refunded (R. 110(3) EPC).

You are reminded that any supplementary search under Article 157(2) EPC will relate only to the last set of claims applicable on expiry of the above time limit AND will be confined to those fee-incurring claims for which fees have been paid in due time.

The fee for the eleventh and each subsequent claim is EUR 45,00.

**Receiving Section** 



Please ignore Epo-form 1226 which was sent to you in error dated 010306.

Ligtvoet, Jan

## PATENT COOPERATION TREATY

#### From the INTERNATIONAL BUREAU

PCT	To:	
NOTIFICATION OF ELECTION  (PCT Article 31(7) and Rule 61.2)	European Patent Office Phoenix Support Help Desk Att. C. Hamm, Room S00G12, P.O. Box 5818 NL- 2280 HV Rijswijk PAYS-BAS	
Date of mailing (day/month/year) 06 May 2005 (06.05.2005)	in its capacity as elected Office	
International application No. PCT/EP2004/051248	Applicant's or agent's file reference TS 5586 PCT	
International filing date (day/month/year) 25 June 2004 (25.06.2004)	Priority date (day/month/year) 27 June 2003 (27.06.2003)	
Applicant SHELL INTERNATIONALE RESE	ARCH MAATSCHAPPIJ B.V. et al	
<ol> <li>The designated Office is hereby notified of its election made in the on:         <ul> <li>17 March 2005 (17.03.2005)</li> </ul> </li> <li>The election</li></ol>		
The International Bureau of WIPO 34, chemin des Colombettes 1211 Geneva 20, Switzerland	Authorized officer  Agnes Wittmann-Regis	
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**EPO Customer Services** 

Tel.: +31 (0)70 340 45 00

Date

01.03.06

Reference TS 5586 EPC P Application No./Patent No.

04766080.8 - 2104 PCT/EP2004051248

Applicant/Proprietor

SHELL INTERNATIONALE RESEARCH MAATSCHAPPIJ B.V.

## Notification of European publication number and information on the application of Article 67(3) EPC

The provisional protection under Article 67(1) and (2) EPC in the individual contracting states becomes effective only when the conditions referred to in Article 67(3) EPC have been fulfilled (for further details, see information brochure of the European Patent Office "National Law relating to the EPC" and additional information in the Official Journal of the European Patent Office).

Pursuant to Article 158(1) EPC the publication under Article 21 PCT of an international application for which the European Patent Office is a designated Office takes the place of the publication of a European patent application.

The bibliographic data of the above-mentioned Euro-PCT application will be published on 12.04.06 in Section I.1 of the European Patent Bulletin. The European publication number is 1644463.

In all future communications to the European Patent Office, please quote the application number plus Directorate number.

#### **Receiving Section**





P.B.5818 - Patentlaan 2 2280 HV Rijswijk (ZH) (070) 3 40 20 40 FAX (070) 3 40 30 16 Europäisches Patentamt European Patent Office Office européen des brevets

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20-02-2006

Reference
TS 5586 EPC P

Application No./Patent No.
04766080.8 - 2104 PCT/EP2004051248 / ISA EP

Applicant/Proprietor
SHELL INTERNATIONALE RESEARCH MAATSCHAPPIJ B.V.

For the aforementioned international application, you are hereby kindly requested to forward to the EPO in its capacity as designated / elected Office:

	a) The publication of the international search report (Art. 20 PCT).	
<b>K</b> I	b) the copy of the international preliminary examination report (Art. 36(3)	)(a) PCT).
	c) the copy (copies) of the priority document(s). If any document is not a EP, please indicate below whether the receiving Office has been required document to the International Bureau (Form PCT/RO/101, Box VI; R.	lested to transmit the
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J	·	2 8. 02. 2006
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Answe	er of the International Bureau [IB]:	•
	The requested item (a), b) or c)] is not available with the IB.  Reason:	
For pr	iority documents [c)] with ISA not EP: The applicant has requested the receiving Office to issue a priority docu Rule 17.1(b) PCT, but the IB did not receive it.	ument [c)] pursuant to

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			Tel.: +31 (0)70 340 45 00
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1 ''	icant/Proprietor ELL INTERNATIONALE RESEARCH	MAATSCHAPPIJ B.\	<b>/</b> .
	the aforementioned international appli apacity as designated / elected Office:		kindly requested to forward to the EPO in
	a) The publication of the internation	al search report (Art. 2	20 PCT).
K	b) the copy of the international prelim	minary examination re	port (Art. 36(3)(a) PCT).
		er the receiving Office	cument is not available and ISA is not the has been requested to transmit the 01, Box VI; R. 17.1(b) PCT).
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Eng	gels, Ulrike		
Ansv	ver of the International Bureau [IB]:		
	The requested item (a), b) or c)] is n	ot available with the IE	3.
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For p	priority documents [c)] with ISA not EP	/• •	
	The applicant has requested the rec Rule 17.1(b) PCT, but the IB did not		a priority document [c)] pursuant to

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# To the European Patent Office



# Entry into the European phase (EPO as designated or elected Office)

European application number	EP04766080.8
PCT application number	PCT/EP2004/051248
PCT publication number	WO05000999
Applicant's or representative's reference	TS 5586 EPC P
1. Applicant	
Particulars of the applicant(s) are contained in the international publication or were recorded by the International Bureau subsequent to the international publication.	
Changes which have not yet been recorded by the International Bureau are set out here:	
Address for correspondence	Shell Internationale Research Maatschappij B.V. P.O. Box 302 NL-2501 CH The Hague Netherlands
2. Representative 1	
This is the representative who will be listed in the Register of European Patents and to whom notifications will be made  Name	
Address of place of business	
Telephone	
Fax	
e-mall	
Any additional representative(s) is/are listed here:	
3. Authorisation	
An individual authorisation is attached.	
A general authorisation has been registered under No:	
A general authorisation has been filed, but not yet registered.	
The authorisation filed with the EPO as PCT receiving Office expressly includes the European phase.	
4. Request for examination  Examination of the application under Art. 94 EPC is hereby requested. The examination fee is being (has been, will be) paid.	✓
Request for examination in an admissible non-EPO language:	Verzocht wordt om onderzoek van de aanvrage als bedoeld in Art. 94.

One or more additional sets of copies of the documents cited in the supplementary European search report are hereby requested.	
Number of additional sets of copies	
5. Documents intended for proceedings before the EPO	
i.1 Proceedings before the EPO as designated Office (PCT I) are to be based on the ollowing documents:	·
the application documents published by the International Bureau (with all claims, description and drawings), where applicable with amended claims under Art. 19 PCT	
unless replaced by the amendments attached.	
Where necessary, clarifications should be attached as 'Other Documents'	
3.2 Proceedings before the EPO as elected Office (PCT II) are to be based on the following locuments:	1
the documents on which the international preliminary examination report is based, including any annexes	
unless replaced by the amendments attached.	
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If the EPO as International Preliminary Examining Authority has been supplied with test reports, these may be used as the basis of proceedings before the EPO.  7. Translations  Translations in one of the official languages of the EPO (English, French, German) are	
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8. Biological material	
The Invention relates to and/or uses biological material deposited under Rule 28 EPC.	
The particulars referred to in Rule 28(1)(c) EPC (if not yet known, the depository institution and the identification reference(s) [number, symbols, etc.] of the depositor) are given in the international publication or in the translation submitted under Section 7 on:	
page(s) / line(s)	
A copy of the receipt(s) of deposit issued by the depositary institution	
is attached	
will be filed at a later date	
A waiver of the right to an undertaking from the requester pursuant to Rule 28(3) EPC is attached.	
9. Nucleotide and amino acid sequences	<del></del>
The items required under Rules 5.2 and 13ter PCT and Rule 111(3) EPC have already been furnished to the EPO.	
The sequence listing as part of the description is attached in PDF format.	
The sequence listing does not include matter that goes beyond the content of the application as filed.	
In addition, the sequence listing data is attached in computer-readable form in accordance with WIPO Standard 25.	
The sequence listing data in computer-readable form in accordance with WIPO Standard 25 is identical to the sequence listing in PDF format.	
10. Designation fees	
10.1 It is currently intended to pay seven times the amount of the designation fee. The designation fees for all the EPC contracting states designated in the international application are thereby deemed to have been paid (Art. 2 No. 3 RFees).	<b>☑</b>
AT BE BG CH&LI CY CZ DE DK EE ES FI FR GB GR HU IE IS IT LT LU LV MC NL PL PT RO SE SI SK TR	
10.2 The declaration in No. 10.1 does not apply. Instead, it is currently intended to pay fewer than seven designation fees for the following EPC contracting states designated in the international application:	
It is requested that no communications under Rule 108(3) EPC be issued in respect of any contracting states not indicated.	
10.3 If an automatic debit order has been issued (Section 12), the EPO is authorised, on explry of the basic period under Rule 107(1)(d) EPC, to debit seven times the amount of the designation fee. If states are indicated in No. 10.2, the EPO will debit designation fees for those states only, unless instructed otherwise before the basic period expires.	

## 11. Extension of the European patent

This application is also considered as being a request for extension to all the non-contracting states to the EPC designated in the international application with which "extension agreements" were in force on the date of filing the international application. However, the extension only takes effect if the prescribed extension fee is paid.

It is currently intended to pay the extension fee for the following states:

#### 12. Debit from deposit account

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Currency

**EUR** 

The European Patent Office is hereby authorised to debit from the following deposit account any fees and costs indicated on the fees page.

Deposit account number

28090005

Account holder

Shell International B.V.

# 13. Reimbursements (if any) should be made to the following EPO deposit account:

V

Number and account holder

28090005, Shell International B.V.

#### 14. Fees

		Factor applied	Fee schedule	Amount to be paid
14-1	005 Designation fee	7	75.00	525.00
14-2	006 Examination fee	1 (40%)	1 430.00	572.00
14-3	015 Claims fee	3	40.00	120.00
14-4	020 Basic national fee for an international application	1	90.00	90.00
	Total:		EUR	1 307.00

#### 15. Annotations

#### 16. Signature(s) of applicant(s) or representative

Place:

Date:

16 January 2006

Signed by:

Subject: NL, Shell International BV, A. Zeestraten 1825

Issuer: , European Patent Office, European Patent Office CA

Capacity:

(Representative)

For employees (Art. 133(3) EPC) having a general authorisation: General authorisation No.



Europäisches Patentamt

European Patent Office Office européen des brevets

## Acknowledgement of receipt

We hereby acknowledge receipt of the form for entry into the European phase (EPO as designated or elected Office) as follows:

Submission number	89504	
PCT application number		
Date of receipt	16 January 2006	
Your référence	TS 5586 EPC P	
Applicant		
Country		
Documents submitted		application-body.xml
	ep-euro-pct.xml	package-data.xml
Submitted by	CN=A. Zeestraten 1825,O=Shell II	nternational BV,C=NL
Method of submission	Online	
Date and time receipt	16 January 2006, 14:54:01	
generated		
Digesi	trans to the contract of the c	:FA:32:C1:DE:60:1D:F0:34:8
	3:5F:9E	

/European Patent Office/



P.B.5818 - Patentlaan 2 2280 HV Rijswijk (ZH) (070) 3 40 20 40 FAX (070) 3 40 30 16 Europäisches Patentamt European Patent Office Office européen des brevets

Generaldirektion 1

Directorate General 1

Direction générale 1

SHELL INTERNATIONALE RESEARCH MAATSCHAPPIJ B.V. Carel van Bylandtlaan 30 2596 HR Den Haag PAYS-BAS



**EPO Customer Services** 

Tel.: +31 (0)70 340 45 00

Date

11,11.05

Application No./Patent No.
04766080.8 - 2104 PCT/EP2004051248

Applicant/Proprietor
SHELL INTERNATIONALE RESEARCH MAATSCHAPPIJ B.V.

#### Entry into the European phase before the European Patent Office

These notes describe the procedural steps required for entry into the European phase before the European Patent Office (EPO). You are advised to read them carefully: failure to take the necessary action in time can lead to your application being deemed withdrawn.

- 1. The above-mentioned international patent application has been given European application No. **04766080.8**.
- 2. Applicants without a residence or their principal place of business in an EPC contracting state may themselves initiate European processing of their international applications, provided they do so before expiry of the 31st month from the priority date (see also point 6 below).

During the European phase before the EPO as designated or elected Office, however, such applicants must be represented by a professional representative (Arts. 133(2) and 134(1), (7) EPC).

Procedural acts performed after expiry of the 31st month by a professional representative who acted during the international phase but is not authorised to act before the EPO have no legal effect and therefore lead to loss of rights.

Please note that a professional representative authorised to act before the EPO and who acted for the applicant during the International phase does not automatically become the representative for the European phase. Applicants are therefore strongly advised to appoint in good time any representative they wish to initiate the European phase for them; otherwise, the EPO has to send all communications direct to the applicant.

- 3. Applicants with a residence or their principal place of business in an EPC contracting state are not obliged to appoint, for the European phase before the EPO as designated or elected Office, a professional representative authorised to act before the EPO.

  However, in view of the complexity of the procedure it is recommended that they do so.
- 4. Applicants and professional representatives are also strongly advised to initiate the European phase using EPO Form 1200 (available free of charge from the EPO). This however is not compulsory.



**Date** 

- 5. To enter the European phase before the EPO, the following acts must be performed. (N.B.: Failure validly to do so will entail loss of rights or other adverse legal consequences.)
  - 5.1 If the EPO is acting as **designated** or **elected** Office (Arts. 22(1)(3) and 39(1) PCT respectively), applicants must, within 31 months from the date of filing or (where applicable) the earliest priority date:
    - Supply a translation of the international application into an EPO official language, if the International Bureau did not publish the application in such a language (Art. 22(1) PCT and Rule 107(1)(a) EPC).

      If the translation is not filed in time, the international application is deemed withdrawn before the EPO (Rule 108(1) EPC).

      This loss of rights is deemed not to have occurred if the translation is then filed within a two-month grace period as from notification of an EPO communication, provided a surcharge is paid at the same time (Rule 108(3) EPC).
    - b) Pay the national basic fee (EUR 160,00) and, where a supplementary European search report has to be drawn up, the search fee (EUR 960,00; Rule 107(1)(c) and (e) EPC).
    - c) If the time limit under Article 79(2) EPC expires before the 31-month time limit, pay the designation fee (EUR 75,00) for each contracting state designated (Rule 107(1)(d) EPC).
    - d) If the time limit under Article 94(2) EPC expires before the 31-month time limit, file the written request for examination and pay the examination fee (EUR 1430,00; Rule 107(1)(f) EPC).
    - e) Pay the third-year renewal fee (EUR 380,00) if it falls due before expiry of the 31-month time limit (Rule 107(1)(g) EPC).

If the fees under (b) to (d) above are not paid in time, or the written request for examination is not filed in time, the international application is deemed withdrawn before the EPO, or the contracting-state designation(s) in question is (are) deemed withdrawn (Rule 108(1) and (2) EPC). However, the fees may still be validly paid within a two-month grace period as from notification of an EPO communication, provided the necessary surcharges are paid at the same time (Rule 108(3) EPC). For the renewal fee under (e) above, the grace period is six months from the fee's due date (Article 86(2) EPC).

- 5.2 If the application documents on which the European grant procedure is to be based comprise more then ten claims, a claims fee is payable within the 31-month time limit under Rule 107(1) EPC for the eleventh and each subsequent claim (Rule 110(1) EPC). The fee can however still be paid within a one-month grace period as from notification of an EPO communication pointing out the failure to pay (Rule 110(2) EPC).
- 6. If the applicant had a representative during the application's international phase, the present notes will be sent to the representative, asking him to inform the applicant accordingly.

All subsequent communications will be sent to the applicant, or - If the EPO is informed of his appointment in time - to the applicant's European representative.



7. For more details about time limits and procedural acts before the EPO as designated and elected Office, see the EPO brochure

How to get a European patent Guide for applicants - Part 2 PCT procedure before the EPO - "Euro-PCT"

This brochure, the list of professional representatives before the EPO, Form 1200 and details of the latest fees are now all available on the Internet under

http://www.european-patent-office.org

**RECEIVING SECTION** 

Date



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## - 2 - **27.** 05. 2005

more carbon atoms is obtained. This bot restriction may be subjected to a catalytic dewaxing treatment.

WO-A-0157166 describes the use of a highly paraffinic base oil as obtained from a Fischer-Tropsch wax in a motor engine lubricant formulation. The examples illustrate that such formulations will also consist of an ester, which according to the description of the patent are added to confer additional desired characteristics, such as additive solvency.

The use of ester co-base fluids in lubricant formulations as illustrated in WO-A-0157166 is not desired because such ester co-base fluids are not widely available and thus expensive. Additive solvency may be improved by using a paraffinic base stock, which contains less paraffins. Such base oils may be prepared by hydroisomerisation of petroleum derived waxes followed by a solvent or catalytic dewaxing step. A disadvantage of such a process is that the starting petroleum derived waxes, such as for example slack wax, are not easily obtainable. Furthermore such waxes may not always have the desired high paraffin content needed to make the desired base oils as per this invention.

US 2003/088133 discloses a process to prepare a base oil from a Fischer-Tropsch derived product by blending the paraffinic base oil with an quantity of C<sub>20</sub>-C<sub>24</sub> alkylcyclohexane in order to improve the additive solubility of the base oil. The alkylcyclohexane compounds are prepared starting from lower boiling Fischer-Tropsch products involving various distillation steps, an alkylation step, a hydrotreating step and a reforming step. A disadvantage of this process is its complexity.

The object of the present invention is to provide a process wherein a base oil with a paraffin content of between 75 and 95 wt% is obtained which does not have the disadvantages of the prior art processes.

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This object is achieved by the following process. Process to prepare a base oil having an paraffin content of between 75 and 95 wt% by subjecting a mixture of a Fischer-Tropsch derived feed and a petroleum derived feed to a catalytic pour point reducing treatment.

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Applicants found that by mixing a relatively small amount of a petroleum derived feed with a Fischer-Tropsch derived feed before performing a catalytic pour point

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